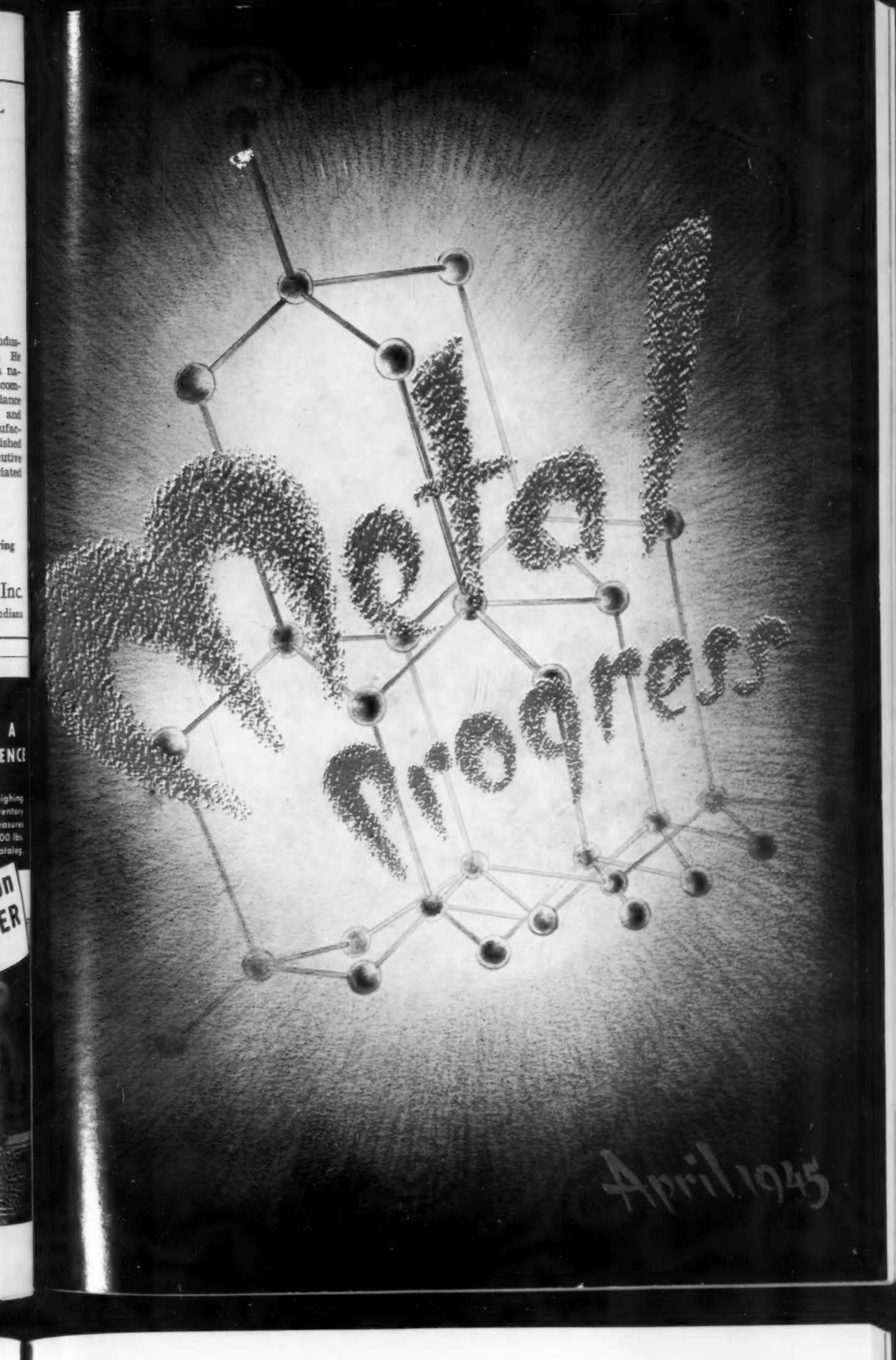
Metal
Progress
April 1945

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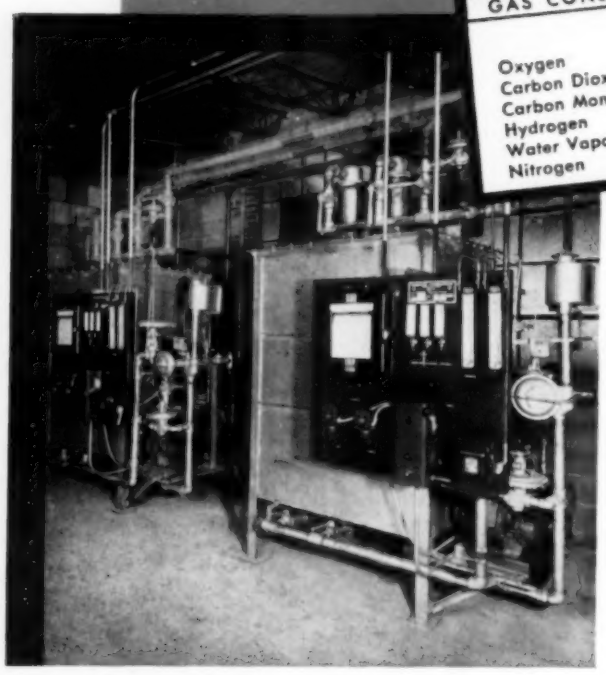
Carbon Monoxide	Carburize	Reduce	------
Hydrogen	------	Reduce	Decarburize
Water Vapor	Oxidize	------	Oxidize-Decarburize
Nitrogen	------	------	------



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METAL PROGRESS

Vol. 47 **April, 1945** **No. 4**

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PREVENTION OF FLAKES IN STEEL FORGING BILLETS

By Paul S. Kingsley
Chief Metallurgist
Follansbee Steel Corp.
Follansbee, West Virginia

THE STEEL DEFECT now commonly called a "flake" was known to exist as far back as 1911 or 1912. The term originated with United States Ordnance inspectors who first noticed them on visual examination of the fractured ends of tensile test pieces. A cooperative study by the U. S. Geological Survey, the U. S. Army Ordnance, the National Bureau of Standards, as well as work by the National Research Council on the nature of this defect, resulted in a general agreement that it was a small particle of steel different in composition from the balance of the piece. The two original publications appear in 1920 *Transactions* of the American Institute of Mining & Metallurgical Engineers, and are by Messrs. Clayton, Foley and Lavey, and by H. S. Rawdon, respectively. It wasn't long, however, until flakes were proven to be discontinuities or ruptures in the metal and efforts were turned to finding their cause.

Studies were made of the part played by internal stresses resulting from the phase changes taking place in the steel during cooling, the weakening effects of segregation, inclusions and dendritic pattern, and the internal pressure exerted by precipitated gases. Each of these by itself, as well as in conjunction with one or more of the others, has been and still is advocated as the prime cause. Notable publications along this line include the one entitled "Flakes and Haircracks in Chromium Steel" by A. Hultgren in the *Journal* of the Iron & Steel Institute for 1925, and another entitled "Iron Oxide and Gas Content of Molten Alloy Steel" by W. A. Hare and Gilbert Soler in *Transactions* for 1937.

An early discovery was that flakes were found to occur only in the cooling following a hot working operation. They can be *experimentally* produced without any immediately preceding hot work, but the necessary conditions are not found in ordinary steel mill operations (rolling and cooling). There is no limiting factor in the usual chemical analysis; flakes can and do occur in almost all grades of high carbon and alloy content.

The necessity that a hot working operation immediately precede the formation of a flake in previously sound steel apparently ruled out the internal stress from phase changes or the weakening effect of segregation as prime causes of the defect. This left the internal pressure from precipitated gases, hydrogen in particular, as the main cause. This conclusion has been developed in such publications as "The Production of Flakes in Steel by Heating in Hydrogen" by R. E. Cramer in *Transactions* for 1937, and "Susceptibility to Flaking and Avoidance of Flake Formations" by E. Klausting in *Metallurgia* for 1937. The explanation was based on the facts that gases diffuse in metals in the atomic state, and that the diffusion of hydrogen in steel increases rapidly with increases in temperature. Also the solubility of hydrogen in steel rises rapidly with increases in temperature over 500° F. This combination of circumstances leads to the precipitation of hydrogen atoms — if present — on cooling, and their combination into molecular hydrogen incapable of diffusing out through the steel, resulting in enough internal pressure to rupture.

Fig. 1 — Etched Slice From 5x5-In. Billet of S.A.E. 4340, Cut After 16-In. Discard From End of Billet Following 136-Hr. Pit Cool. Heat tested 11/26/43 and all tests were free from flakes

Fig. 2 — Etched Slice From Same Heat as Fig. 1, Cut After 16-In. Discard From End of Pit-Cooled Billets After Being Stocked Six Months. Tested 5/18/44; 60% of the billets were flaked

This theory was further supported by the only method then known of successfully preventing flakes — slow cooling following all hot working operations. The hydrogen was supposed to diffuse out of the steel during slow cooling and, once having escaped or established escape paths, no internal pressures could be built up in subsequent heating and cooling operations, unless these escape paths were closed by hot deforming the material. Hence the requirement that hot work was a necessary preliminary to the slow cooling (during which the flakes actually formed).

Largely due to studies at University of Illinois on internal transverse fissures in rail steel as well as shatter cracks (the railroad man's name for "flakes") the production of sound rails has been put on a firm basis. Consequently the publications from these workers have unusual authority. Reference may be made to "The Production of Flakes by Treating Molten Steel With Hydrogen, and the Time of Cooling Necessary to Prevent Their Formation", and to "The Prevention of Flakes by Holding Railroad Rails at Various Constant Temperatures", both by R. E. Cramer and E. C. Bast in *Transactions* for 1939.

Numerous investigations are still in progress on the effects of hydrogen (see "Flaking in Alloy Steel" as discussed in the 1944 meeting of the Openhearth Committee, A.I.M.E.) and a large number of metallurgists, particularly the research workers, would have us believe that it is the prime cause of flakes. Frequent references to segregated areas are an admission that there is *something* in steel, a necessary prior cause, in addition to hydrogen, even though there has been no success in any of the attempts to correlate segregation as measured by the known causes, such as mold size, pouring temperature, and rate of rise in the mold, with susceptibility to flaking.

The known facts about flaking are as follows: Large size pieces are more prone to flakes than small pieces; some analyses, high in carbon and alloy content, are more prone to flakes than lower analyses; flakes are never encountered in the austenitic stainless grades, even though they contain sufficient hydrogen to cause bleeding of the ingots during solidification; heats of the same chemical grade have different susceptibility to flakes, occasional heats being free from flakes on air cooling and companion heats made and rolled the same way being extremely sensitive to flake formation; occasional heats, which are slow cooled from rolling and are free from flakes when tested, will develop flakes at a later date. Apparently some heats require a definite period of germination after the material reaches room temperature before the ruptures occur; an example is seen in Fig. 1 and 2 and their captions.

A. O. Bamberger has described, in an article in *Iron and Steel Engineer* for November 1943 entitled "The Prevention of Flakes", an attempt to combine the two time periods required for *(a)* slow cooling of forgings to prevent flakes and *(b)* a subsequent required heat treating operation. This has resulted in a method for preventing flakes radically different from previously accepted procedures. This cycle consists of: *(a)* cooling in air to below the transformation temperature but not low enough for any hydrogen precipitation; *(b)* reheating to above the Ac_1 temperature, with a long enough soak to assure uniformity of temperature, and *(c)* air cooling. Various modifications of this cycle have been attempted, such as holding at various temperatures for isothermal transformation without the subsequent reheating, cooling to temperatures above Ar_1 and then reheating, or cooling to below Ar_1 and reheating to below the Ac_1. Some of these modifications have met with success, but more often the results have been unsatisfactory.

It should be stated that this successful cycle cooling method for preventing flakes cannot be adequately explained by the hydrogen theory alone. The material on the first cooling is not allowed to go low enough in temperature for any large precipitation of hydrogen, so this gas must still be in solution at the second air cooling, whereupon it is precipitated — but no flakes result.

The author's theory of flake formation is based on the following considerations: Flakes are internal ruptures, obviously caused by stress greater than the strength of the material, and are the resultant of several individual stresses:

1. A thermal stress of contraction caused by the temperature gradient from the outside to the inside of the piece.
2. A stress caused by pressure of precipitated hydrogen.
3. A stress caused by the phase changes during cooling.
4. A residual stress remaining from the hot working.

When steel is slow cooled, all of these are partially relieved and — what is possibly more to the point — reach their maximum intensity at different times. Furthermore, the variation in susceptibility to flakes found in different heats is due to the difference in the amount of hydrogen in solution.

When steel is cycle cooled, the first air cooling to below the Ar_1 point establishes the thermal stress and the stress due to phase change, without allowing any hydrogen to precipitate. The subsequent reheating relieves the phase change stress and the residual stress left from the hot working. In the final air cooling, only the thermal stress and the stress due to hydrogen precipitation are generated. The resultant of these two is not sufficient to rupture the material.

In heats which are rolled to both large and small sizes it is often found that the large billets show flakes while the smaller sizes are satisfactory. The thermal stress in the large size is great enough to give a resultant combined stress which will cause rupture, while in the smaller size this part of the total stress is low enough so that no flakes result. None of these variables is encountered in cycle cooling, since the stresses are never allowed to generate all at the same time.

Recommended Cycles

Using the above theory of cycle cooling, a thermal treatment has been devised and used successfully by several steel companies to prevent flakes in alloy billets. These cycles always cool the billets so that *all* of the steel is below the Ar_1 temperature. Unless this cooling practice is

Table I — Heating Cycles to Avoid Flakes in Steels
All temperatures are actual steel temperatures

Grade	Carbon	Manganese	Silicon	Nickel	Chromium	Molybdenum
Cycle I — Rapid cool to 800°/700°, and even out with hold for specified time as determined by billet size. Reheat to 1250°/1300° and hold for specified time. Furnace cool to 1200° and air cool.						
Ni-Cr-Mo	0.30/0.50	0.50/0.80	0.15/0.35	1.00/2.00	0.50/0.80	0.20/0.40
Cycle II — Rapid cool to 750°/650°, and even out with hold for specified time as determined by billet size. Reheat to 1250°/1300° and hold for specified time. Furnace cool to 1200° and air cool.						
5% Ni	0.10/0.20	0.30/0.60	0.15/0.35	4.75/5.25	—	—
3½% Ni-Mo	0.10/0.30	0.40/0.60	0.15/0.35	3.25/3.75	—	0.20/0.30
Cycle III — Rapid cool to 1200°/1100°, and even out with hold for specified time as determined by billet size. Reheat to 1300°/1350° and even out with hold for specified time. Furnace cool to 1200° and air cool.						
52100	0.90/1.10	0.30/0.60	0.15/0.35	—	0.90/1.50	—
52100 + Mo	0.90/1.10	0.30/0.60	0.15/0.35	—	0.90/1.50	0.20/0.40
9255, 9260	0.50/0.65	0.70/1.00	1.80/2.20	—	—	—
4100	0.30/0.50	0.70/0.90	0.15/0.35	—	0.60/1.10	0.15/0.25
5100	0.30/0.50	0.70/0.90	0.15/0.35	—	0.70/0.90	
9262	0.55/0.65	0.75/1.00	1.80/2.20	—	—	—
Cycle IV — Rapid cool to 1100°/1000° and even out with hold for specified time as determined by billet size. Reheat to 1375°/1425° and hold for specified time. Furnace cool to 1200° and air cool.						
3100	0.30/0.50	0.60/0.90	0.15/0.35	1.10/1.40	0.55/0.90	—
3200	0.30/0.40	0.30/0.60	0.15/0.35	1.65/2.00	0.90/1.20	—
3300	0.10/0.35	0.30/0.60	0.15/0.35	3.25/3.75	1.40/1.75	—
3400	0.10/0.40	0.30/0.60	0.15/0.35	2.75/3.25	0.60/0.95	—
4600	0.20/0.50	0.60/0.80	0.15/0.35	1.65/2.00	0.20/0.30	—
NE8600	0.15/0.30	0.70/0.90	0.15/0.35	0.40/0.60	0.40/0.60	0.20/0.30
NE9400	0.15/0.50	0.80/1.20	0.40/0.60	0.20/0.50	0.20/0.40	0.08/0.15
NE9500	0.30/0.50	1.20/1.50	0.40/0.60	0.40/0.70	0.40/0.60	0.15/0.25

adhered to, flakes often result. The steel may be air cooled as rapidly as possible, but must be held below Ar_1 long enough so that the center of the lift will reach the required temperature. Care must be used that the ends of the billets and the outside billets of the lift do not cool below the danger point of 500° F., where ruptures will be caused by hydrogen precipitation. The billets are then heated to a temperature just below the Ac_1 point. This temperature is used so that no phase change will take place on the subsequent air cooling, and the steel will be as soft as possible within the required thermal limitations of this treatment. Since the critical temperatures of a number of the various alloy grades are reasonably close together, they can be grouped and the number of required cycles reduced materially.

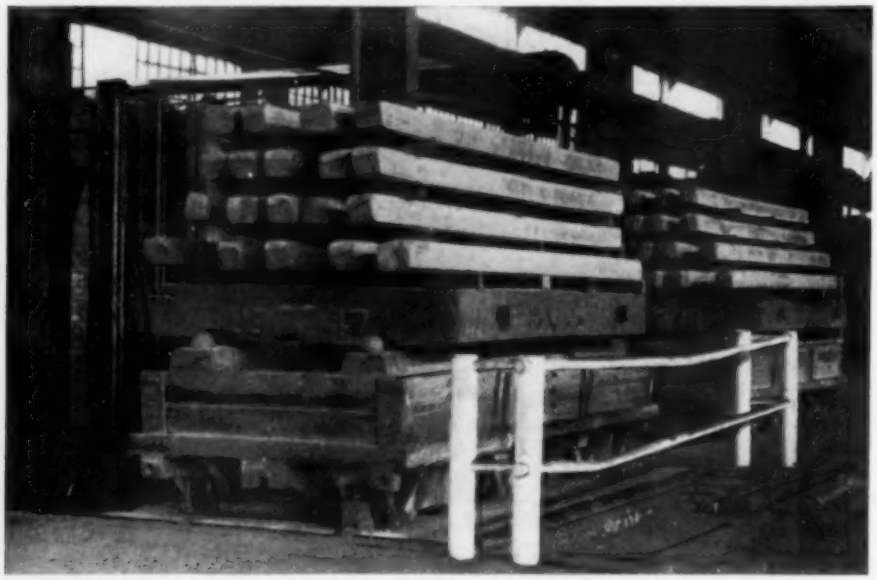

Fig. 3 — Car Loaded With Billets, on Transfer Track Ready for Entrance Into Ante-Chamber at Beginning of Dressler Kiln

In fact four cycles will be found to include the necessary conditions for all the heavy forging steels now in production. These are listed in Table I, page 701.

Some provision is required for variation in billet size, since smaller billets will cool or absorb heat faster than larger ones. Holding times should therefore be specified at the lower temperatures, for assuring that the center of the lift reaches the specified temperature. Soaking times at the reheating temperatures are also set up, based on billet size and for lifts averaging 40,000 lb. It has been found that billets up to and less than 6×6 in. in size may be cycled the same as 6×6's; larger billets require longer times. (The largest billet in the furnace determines the time.) Specific recommendations are as follows:

Size	Hold in Lower Temperature Range	Hold in Higher Temperature Range
6 x 6	6 hr.	6 hr.
7 x 7	6	8
8 x 8	6½	8½
9 x 9	7	9
10 x 10	8	10
11 x 11	8½	10½
12 x 12	9	11

These cycles were set up to take billets ranging from 3½×3½ to 12×12 in. as delivered from the blooming mill shears, and treated in a sheet annealing furnace of the box type. The billets were loaded on the lift bottoms with the first layer supported on rail sections and each succeeding layer separated similarly. The billets were spaced apart, side by side, for free circulation of air during cooling and of furnace gases during reheating. During the development of the cycles, thermocouples were placed on the bottom in the center to control the temperatures of the billets. Now that the cycles have been thoroughly tested, time of holding has been found to be an adequate control and a furnace thermocouple controls the reheating temperatures. When the ends of the billets and the outside billets drop down to the specified temperature, the bottom is pushed into the furnace and the doors closed. The heat given off by the warmer center billets in the lift evens out the temperature of the entire lift without letting the outside billets become cold enough for flakes to form.

Continuous Kiln for Furnace

The prime purpose of cycle cooling is a greater assurance of freedom from flakes and a faster operation. Slow cooling normally consumes in excess of 96 hr.; with some pits it requires as much as 170 hr. Cycles in these box furnaces range from 30 to 70 hr., depending on the billet size. In order to reduce this time and also to cut the cost, two 345-ft. Dressler kilns originally built for the continuous annealing of

Fig. 4 — View Down Tunnel of Furnace at the Beginning of the Soaking Zone, Showing How Cars Form the Furnace Bottom. In the cooling end of the furnace the walls are lined with a series of large diameter steel pipes, placed horizontally

sheet steel, were converted to the continuous cycle cooling of alloy billets.

Billets are piled on annealing trays, which measure 66×180 in., as they are delivered from the mill, cut to the ordered length. Railroad rail sections are put on the trays first and in between the layers of billets, as was done when cycle cooling in box furnaces. Limits have been set up for the number of billets in a layer and for the number of layers in order to insure uniformity during cooling and reheating. These range from 12 billets to a layer with 5 layers for 4×4's, to three billets to a layer with three layers for 12×12's. After the lift has cooled to the specified temperature, as measured by "Tempil Sticks" and visual examination, the tray is loaded on a car (Fig. 3).

This car, when put into furnace, forms part of a train whose deck serves as the bottom of the furnace. The train is composed of 21 cars, and before a car can be pushed into the furnace at the entrance end, a car must be taken from the delivery end.

The 345-ft. furnace is divided into three zones — the first zone or preheating section of 120 ft., the second or soaking section of 60 ft., and the third or cooling section. The furnace (see Fig. 4) is heated by five North American burners for natural gas on each side of the soaking zone. These burn in tile tunnels on each side of the furnace with the flame and waste gases traveling along each side parallel to the length of the furnace toward the entrance end. The stack, which is located at the entrance end, provides the

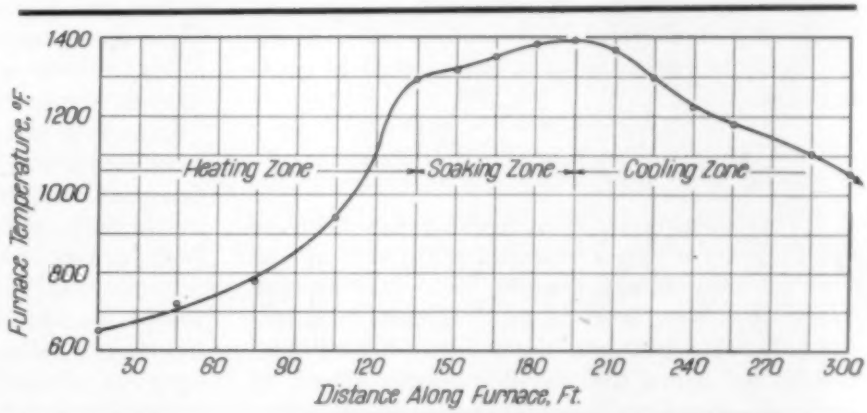

Fig. 5 — Thermal Profile for Charges of 7x7-In. Billets of S.A.E. 4340 Steel, Charged 11:30 A.M. 11/11 and Removed 5:30 A.M. 11/13; Cars Pushed Every 2 Hr. Ac_1=1350; Ar_1=725° F.

necessary draft. In this way the work is heated during the entire time the car is traveling from the entrance through the soaking zone.

In the cooling zone, heat is extracted by drawing atmospheric air through steel tubes along the walls. This air is used for combustion after being further pre-heated. Figure 5 (page 703) is a temperature profile of the furnace when treating 7×7-in. billets of S.A.E. 4340 steel.

In addition to these three zones in the furnace proper, there are two ante-chambers, one at each end, for the admission and discharge of the cars. These are fitted with inner and outer doors, so a car may be charged or discharged without opening the tunnel to the outside atmosphere. This allows a much closer control of combustion and temperature within the furnace.

The rate of rolling on the mill, the size of the billets, and the amount placed on each tray control the rate of cooling of the lift and how often cars can be put into the furnace, since the billets must cool to below the specified temperature before they can be removed to the outer air. Our experience has been that the furnace can heat the billets fast enough so that we can keep up with mill production. Very often we have five or more cars of hot billets, ready to enter the furnace. To arrive at some uniformity, we have set up limits of heating by establishing a minimum time in the soaking zone for various size billets, as follows:

Billet Size	Minimum Time in Soaking Zone
3½ to 5 in. square	2½ hr.
5 to 7½ in. square	3¼
7½ to 12 in. square	4½

Nothing under this time is permitted — although it may be exceeded, since the temperature in the soaking zone is under the Ac_1 point and time at temperature is not a factor. Bury pits and box furnaces are available to take care of the hot steel from the blooming mill, should any delay be encountered in the continuous cooling zone.

Results: Flake-Free Steel

This continuous cycle cooling has been in operation for nearly a year and out of over 25,000 tons of alloy billets treated, only two lifts of flaked steel have been found. (In both of these, the lifts were put into the furnace before they had cooled below the specified temperature.) It has been found to be much less susceptible to errors on the part of the operators, to deliver steel somewhat softer than that obtained from box furnace cycle cooling, and to be cheaper to operate than the box type of furnace.

CRITICAL

SURPRISED to learn of a rather widespread and growing sales resistance to the National Emergency steels on the basis that they are now contaminated with "battlefield scrap" and will therefore be unreliable — this coming from men who have used these same steels successfully in the past and recognize their success in absorbing usefully the valuable alloying metals in circulating home scrap. Just how a modicum of nickel or chromium or molybdenum recovered from a tank transmission that was shot up in Normandy can be any different from the alloy reclaimed from a duplicate part rejected in Toledo by an eagle-eyed inspector is hard to imagine. Maybe the news has got about that brass mills do not like to put more than 15 or 20% of fired cartridge cases into their melting pots when making more cartridge brass, and Uncle Sam's mint is therefore using some of them instead to manufacture the lowly penny. Maybe this is how the purchasing agent's mind works: "If fired cartridge cases from the battlefield can't make good new cartridge cases, then *steel* scrap from the battlefield can't make good new steel." If so, it will take some telling and retelling to tell him that the one conclusion doesn't follow from the other. Fired cartridge cases contain about 1% antimony sulphide in their primers, and this is not eliminated in the simple melting of a brass ingot, to the detriment of its ability to withstand heaviest cold working in the drawing presses. Steel manufacture, however, as all certainly must know, is done under refining conditions, and all manner of dirt is slagged off from the scrap and pig iron charged. Anyone who has taken a look at the "country scrap" the American steel industry was forced to use during 1943, and from which very satisfactory steel indeed was made, will agree that the dirtiest battlefield should yield a cleaner charge. As a matter of fact, munitions are made of much more carefully refined, processed and inspected metal than the average of peacetime goods, and it's a good bet that a steel maker would gladly change

Battlefield scrap — steel and brass

POINTS

from scrap automobiles that never left the country to scrap tanks or guns that received mortal wounds on foreign fields.

REMEMBERED how in the early days of Hitler's war it was freely predicted that he could not last long because he had such limited supplies of money, oil and alloying metals. So was greatly interested in learning from officers on Gen. G. M. BARNES' staff that Army Ordnance Department has not found until very recently any evidence that the Germans have *insufficient* strategic alloying metals or satisfactory substitutes. The incorrect predictions about other materials were based on Germany's limited or non-existent production and the belief that imports could be blockaded.

Small lack of alloys in Germany

Error came through neglecting the fact that the Germans had accumulated stockpiles of everything the high command thought the armies would need, and that they later captured in France and Czechoslovakia similar generous stockpiles, either as such or worked up into guns, armor and tanks — metals that were promptly reworked. Furthermore, they had no compunctions about combing their conquests for the last scrap of desirable metal; much manganese ore also came in from Russia and chromite from Greece and Turkey. As the war progressed, there has therefore been very little change in the nature and treatment of the alloys going into German munitions. Things like guns and engine parts that needed hardenability got a sufficient amount of molybdenum and chromium — in some instances even more than we would have thought necessary. Nickel, which has always been short, has been reserved for aircraft and other most essential purposes. German tank armor has considerably higher carbon and lower alloy than ours, which is responsible for its lower toughness and poorer weldability, counterbalanced by the frequent emergency festooning of extra armor plates or track treads about the more vital areas of the panzers.....Knowing armament as we know automobiles, it is unlikely that the Germans would use anything but excellent steel making and steel treating practices — and this is proven by examination of much captured matériel. (See the notable study of the subject by War Metallurgy Committee in the February issue of *Metal Progress.)*

Iron powder for shell rotating bands

One interesting substitution for copper, the most critical of all metals in Germany, is in rotating bands on projectiles. The "standard" metal is copper or gilding metal (10% zinc). The Germans have found that bands made of iron powder are satisfactory. Chemical analyses indicate that low carbon scrap may have been the ultimate raw material, for the iron band has about the same content in manganese and carbon, as well as ordinary amounts of "tramp" alloy. Many cartridge cases in all calibers are made of steel, but not to the entire exclusion of brass. As shown in the leading article in the March issue, the steel used is a low carbon, non-aging variety, ordinarily completely spheroidized in microstructure. Rust protection is either by copper or brass plate inside and out or, better, by phosphate treatment covered with lacquer.

REMEMBERING LLOYD GEORGE's dictum that war is entirely too important a matter to be left to the generals, and exercising the right to be an armchair strategist in a war which is so intensely personal to every man, responded to Baltimore Chapter's invitation with a talk on "Metals in Armament (With Some Thoughts on Influence of Air Power on Warfare)" — mostly the latter. Many happenings in this war have already refuted the predictions of over-enthusiastic air men that the doom of battleships has been struck — to say nothing of the other weapons of World War I. This new war is now old enough so that it should now be in order to attempt to strike a balance between some of the rash predictions and sober fact.....

The future of warplanes

Of the various functions of warplanes, the one where it seems to stand pre-eminent, in the sense that it does something never done before, is for transporting explosives, men and materials for long distances and rapidly over formerly insurmountable obstacles and defenses. This does not mean — even after making reasonable allowances for future improvements in aircraft — that the airplane will be the *only* carrier, and there will be no future use for ships to ply the oceans, or for

By the Editor

railroads and motor cars to traverse land, or artillery to fire explosive shells. For the airplane to supplant the battleship, for example, it would require that the airplane be able to transport *all* necessary men, matériel, and supplies for an invading army, else an opponent with well protected naval power could prevent auxiliary supplies from arriving afloat. Now it is more than doubtful that the airplane can do so.....A long conference with some of GLENN MARTIN's engineers did nothing to shake this conclusion. We attempted to predict the range, carrying capacity and speed of tomorrow's air transport by estimating the effect of improvements reasonably to be expected in materials, in power plant, and in aerodynamic design; while it is within reason to expect that all *troops* would be airborne in the last lap of an invasion or campaign, there is no justification for expecting that the cost of flying *freight* (now 7 to 8¢ per ton-mile) will ever approach that of floating the cargo (about 0.2¢ per ton-mile). Since these costs ultimately reflect the man-hours required in preparing and operating the respective services, and since our only serious lack in this war is man-hours, there seems little chance that we could ever afford such extravagance in a struggle for life and death.....Of the other two principal functions of warplanes — reconnaissance and bombardment — while they already excel the older devices, they are in turn being pressed by new weapons like radar for observation and rockets for long-range projectiles. If flying bombs, "vengeance weapons" and similar devilish devices are classed as aircraft rather than projectiles, then one hesitates to project his mind ahead, appalled with the thought that unless adequate defense or preventive measures can be devised, man — the animal — is on the road to self-destruction by creating an artificial environment in which no animal can live.

Freight — flown or floated?

ABSORBED, at GLENN MARTIN's large plant near Baltimore, some of the enthusiasm found there for some new 75S aluminum extrusions, with 78,000-psi. yield and 90,000 ultimate — figures that indicate it might replace normalized steel in aircraft parts, in the opinion of C. G. STEPHENS, inspection manager, and currently chairman of the Baltimore Chapter. (He also exhibited some extrusions of record size — about 24 sq.in. in cross-section, 50 ft. long, weighing 750 lb.). The possession of a single alloy of such excellent properties does not mean that the entire airplane will be forthwith transformed, for he tells me that the large "Marauders" and "Mariners" now in production contain 26 different aluminum alloys (15 wrought and 11 cast), 35 steels, mostly alloy, 11 corrosion resistant high alloys, 14 brasses and bronzes, and four magnesium alloys. This is in the structure alone, and says nothing of the fabrics, plastics and rubbers, all of which together give interest to the life of a materials engineer. The list could be simplified, and undoubtedly will be when there is time; many of the steels could be replaced by 4340, "which has everything", says STEPHENS.....Interesting and exhaustive studies are under way, directed toward the application of spot welding to structural members of aluminum. Seemingly the largest variables in most welding machines stem from the devices for exerting pressure on the electrodes — the electrical control is far more precise. Pending better results in production from spot welding, all stressed skin on Martin ships is riveted to the frame. To insure against a dimple where a countersink may be too large or the rivet driven too hard, the rivets are driven with a slight surplus of metal in the outside head, and the extra metal cut off with a light hand tool that operates like a little lawn mower, the cutter blades rotating at high speed and clearing the general surface by 0.003 in. Apparently this small projection left on the rivet head causes less turbulence in the slip stream than a small dimple from an improper countersink.....Two curious notes: *(a)* "Nylon" tires *grow* at a variable rate for two days after first inflation and sometimes don't fit very well in the retractable gear. *(b)* While stainless or inconel is excellent for exhaust collector rings on radial engines, individual exhaust stacks are better made of carbon steel sheet, protected with porcelain enamel.

Galaxy of metals in airframes

Lawn mower for rivet heads

ACCORDING TO *Army Ordnance Bulletin*, America's Industry-Ordnance team has produced in the 4½ years through 1944 about 68,000 tanks, 2,800,000 large and medium guns, 15,000,000 machine guns and rifles, 43,000,000,000 rounds of ammunition (of which 35,820,000,000 rounds were for small arms) and 43,400,000 bombs, to say nothing of other types of motorized equipment and an amazing diversity of auxiliary material — in all some 1200 major items of ordnance. About 100,000 barrels for large caliber guns and howitzers have been made of centrifugal castings; pierced billets account for another sizable number.

Arsenal of democracy

FAILURE OF SPRING LOOPS BY STRESS CORROSION

By Given Brewer
Stress Analyst
and Herman C. Ihsen
Metallurgist
Consolidated Vultee Aircraft Corp.
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An example of the mis-treatment of spring wire, all too common in commercial products, and especially prevalent in cold wound springs made of oil-tempered wire and then cadmium plated. The thing that is likely to puzzle some is the location of the cracks, namely on the inside of the spiral or end loops, but elementary stress analysis clearly shows the reason therefor.

RECENTLY a large manufacturing concern found a shipment of several thousand spring clips, of the type shown in Fig. 1, page 708, to be defective. It was discovered that the looped ends of the wire could be twisted off with very little force and subsequent microscopic examination of the looped ends revealed the presence of radial cracks in that location. These cracks were unusual in the respect that they emanated from the *inside* surface of the loop (see Fig. 2), whereas the familiar type of crack resulting from an over-severe forming operation always occurs at the *outside* surface of the bend. The springs were 5½ in. overall length, formed of 0.068-in. oil-tempered wire, and the inside radius of the end loop was 0.060 in.

Detailed inspection over the entire length of the spring disclosed the fact that the cracking occurred only at the looped ends and was entirely confined to the inner surface of curvature. As the springs from this shipment were useless for service it was a matter of first importance to discover the cause of the cracking and take prompt preventive actions.

In order that a rational analysis of the failures might be made the prior history of the springs was determined, including the various processing treatments to which they had been subjected as well as the chemical composition and the physical properties of the wire. The wire was found to be oil-tempered S.A.E. 1065 steel having an ultimate tensile strength of 225,000 psi. The springs had been cold formed from the wire in the heat treated condition, the first operation being to form the central saddle section; the final operation was to form the end loops. After the forming operation the springs were "stress relieved" at 500° F. and then given two coats of primer before shipment by the manufacturer.

Shortly after their arrival at the customer's plant the springs were rejected because of faulty paint adherence. To salvage the shipment it was sent to the metal plating department for cleaning and repainting. In the customer's plating department the springs were processed as follows:

1. Immersed in a caustic electrolytic cleaning compound for a period between 5 and 10 min.

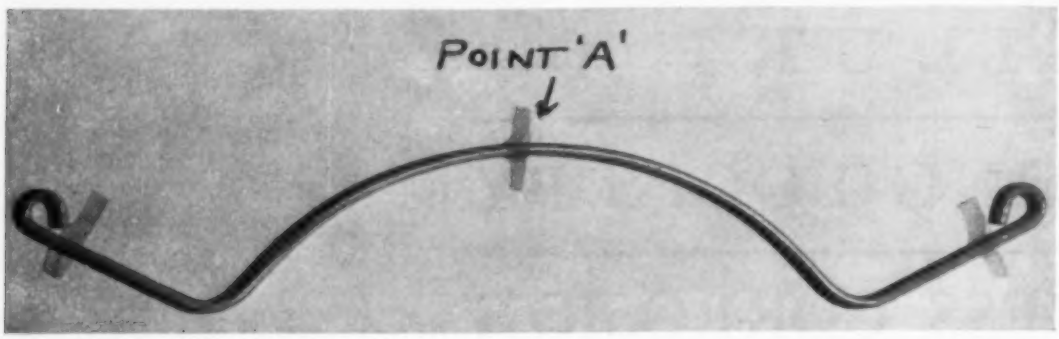


Fig. 1— Unsatisfactory Springs, Full Size

2. Immersed in a 10% solution of hydrochloric acid for a period between 5 and 10 min., washed in water and dried.

3. Cadmium plated.

4. Baked at 350 to 400° F. for relief of hydrogen embrittlement.

5. Looped ends painted.

When these springs were used in assembly the greatest number broke at the looped ends during installation.

In view of the past history of the springs and on the chance that the embrittlement might be due primarily to "hydrogen embrittlement", several of the clips were furnace heated to various temperatures up to 1450° F., but this failed to alleviate the brittle condition of the loops. At this juncture the polished sections of the loops were examined and the microscope revealed the presence of radial cracks emanating from the inside surface of the loops (Fig. 2). It became obvious that nothing could be done to salvage the springs, and efforts were then directed toward a complete solution of the problem to prevent its recurrence.

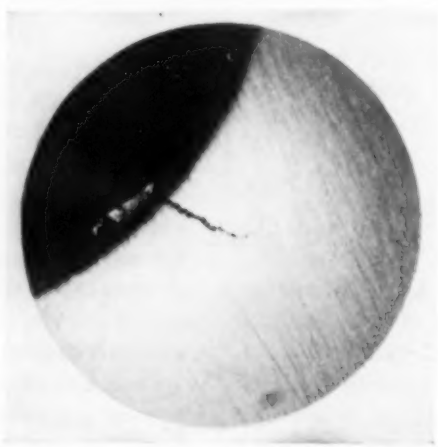


Fig. 2 (Above) — Unetched Microsection Showing Crack (One of Several) Starting From Inside of End Loop, Magnified 40×

In an earlier article by one of the present authors on the subject of residual stresses in bridge wire loops (*Metal Progress*, September 1943, page 441), it was shown that although high tension stresses beyond the yield strength of the material exist at the outside surface of a bend during the forming operation, the tension stresses on the outside of the bend give way to residual compression stresses during recovery or spring-back and at the same time the inside of the bend (initially under compression) retains tension stresses after recovery. A sketch of external conditions during forming around a pin and after spring-back is shown in Fig. 3, and an approximation of the residual stresses after spring-back is shown in Fig. 4. (The latter is an adaptation of an illustration in the article in *Metal Progress;* interested readers should refer to it for a quantitative demonstration of the derivation of the stress conditions in round wire after forming and spring-back.)

As the loops of the defective spring clips had been cold formed it was possible that the residual tension stresses at the inner surface of the loop due to spring-back had not been removed by the 500° F. stress relief at the manufacturer's plant. If the high residual tension stresses were still present at the inner surface of the loop it was reasoned that the radial cracks in this same region could have been caused by stress corrosion or by hydrogen embrittlement during one or more of the processing operations already

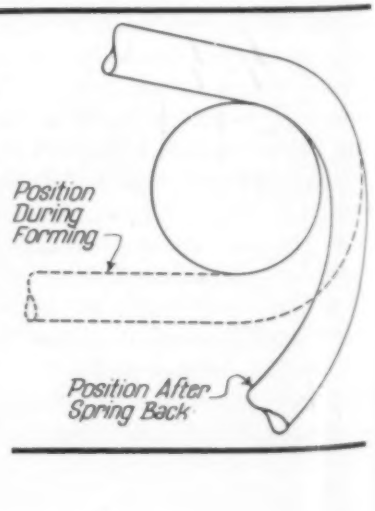


Fig. 3 — Conditions Existing During Forming of Wire Around Small Pin, and After Spring-Back

enumerated. To check this possibility of stress corrosion attack and to isolate the offending electrolyte, if it existed, a number of the spring clips were selected and looped at Point *'A'* in the undamaged center of the spring (see Fig. 1 and 5). It should be noted that the cadmium plating was not removed from these clips which were looped at the center. The loops, as formed, had approximately the same radius as the original end loops but the center loops could be straightened out without failure or other evidence of embrittlement. A microscopic study of the wire, sectioned at the central loop in various ways, showed no evidence of cracking, and confirms the belief that the damaging cracks at the end did not occur as the result of the mechanical looping operation.

Fig. 5 — Spring After Loop Had Been Wound at Point A of Fig. 1

The remainder of the springs having the extra loops in the center were grouped and then subjected to the following processing operations:

Group A — Processed in the same manner as the original springs — that is, immersed in caustic electrolytic cleaner, held 5 to 10 min., then immersed in a 10% solution of hydrochloric acid for a period of 5 to 10 min., washed and dried.

Group B — Processed only in the caustic electrolytic cleaner.

Group C — Immersed only in the 10% hydrochloric acid solution.

All three groups were then tested for embrittlement by straightening open the middle loops. It was found that the springs in Groups A and C were embrittled; they broke at the loops under a small applied force. This brief experiment isolated the hydrochloric acid pickle as the electrolyte in which the corrosive cracking occurred. Micrographs of the center loops of the three groups after processing are shown in Fig. 6.

The presence of radial cracks in the micrographs taken of typical sections from Groups A and C further corroborated the evidence at hand, namely that the wires failed by stress corrosion at the point of maximum residual tension stress while in the 10% hydrochloric acid pickling solution. Of incidental interest was the fact that the springs cracked, despite the cadmium plate.

To determine the effect of "stress relief" at various annealing temperatures a number of additional samples were prepared by forming loops in the central portions of the spring clips in the manner already described. After the looping operation the springs were furnace heated to various temperatures, 950, 1200 and 1450° F., following which the clips were pickled in the hydrochloric acid solution. The results obtained were as follows:

Stress Relief Temperature	Loops Tested	Condition of Loops After Pickling	
		Ductile	Brittle
950° F.	6	4	2
1200	7	5	2
1450	7	6	1

On the basis of this limited study it would seem that the range of temperatures commonly assumed to relieve internal stresses are not effective in the case of high residual stress. At least there can be no assurance that cracks will not occur during pickling operations on heat treated but cold formed springs, despite "stress relief" at temperatures below those that would anneal it ("destroy the temper" of the wire).

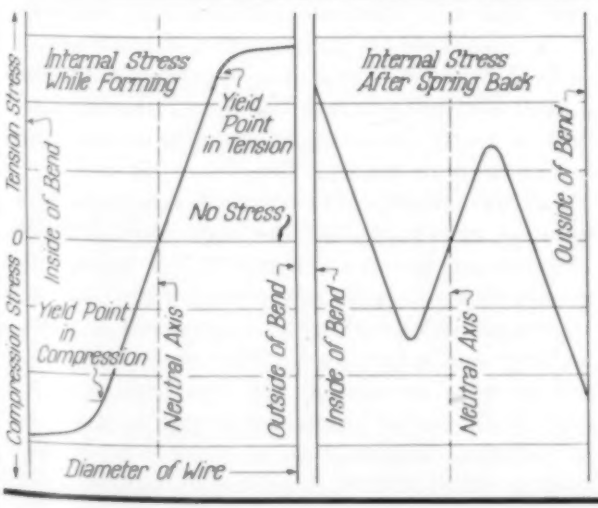


Fig. 4 — Approximate Internal Stress Distribution in Wire While Forming Around Small Pin, and After External Forces Are Released Allowing Spring-Back (Loop "Lies Dead"). Note the large residual tensile stresses at inside of bend

Significantly the manufacturing company in this instance stated that it was its policy to prohibit the acid pickling of heat treated springs after cold forming, but that it had been its experience that cold drawn spring wire, cold formed into springs, could be acid pickled with impunity. Corroborating this shop practice, R. R. Tatnall, writing in *Metals and Alloys* for January 1944 on the subject of hydrogen embrittlement, states that heat treated 1065 wire is far more sensitive to hydrogen embrittlement in acid pickle than is cold drawn wire of the same analysis, and that sometimes 15 sec. in the pickling solution will cause failure in the heat treated wire.

The spring company submitted a number of strands of 0.072-in. cold drawn 1065 steel wire having an ultimate tensile strength between 233,000 and 240,000 psi. This wire was looped in the same manner as was the heat treated wire previously described, and then placed in the acid pickling solution for various periods of time up to and including 15 min. After the loops were washed and dried microscopic examination of the bends failed to reveal any evidence of cracking. On the other hand, looped samples of the heat treated wire placed in the same solution were found to have developed cracks in 2 min.

The superior resistance to stress corrosion inherent in the 1065 cold drawn wire as compared to the 1065 oil tempered wire of the same ultimate tensile strength is of particular significance. The greater susceptibility of heat treated wire to hydrogen embrittlement — as compared to cold drawn wire of equivalent tensile properties and chemical analysis — is well known throughout the wire and spring industry, yet is evidently not so well known to metallurgists generally. A. V. de Forest, professor of mechanical engineering at Massachusetts Institute of Technology, informs us that troubles frequently are brought to his attention that might readily have been avoided if due regard had been paid to the different characteristics of these two classes of material, outwardly almost indistinguishable. For example, a spring may be made by winding oil-tempered wire over a mandrel and then held in restraint, allowing only the slightest spring-back; in such a spring even without pickling and no question of hydrogen embrittlement, cracks will sometimes form on the *outside* of the bend (in heavy tension, as shown in the left-hand part of Fig. 4). Such failures are usually associated with a poor surface condition, even without active corrosion to set up a dangerous "stress raiser" at a tiny notch. The same type of breakage occurs in watch and clock main springs made of flat strip, which are held in keepers to prevent anything more than partial spring-back, but the stress raising defect here is usually located on the edge of the spring rather than on the flat.

This difference in surface condition is of greatest importance in the serviceability of the two classes of wire. Hard drawn spring wire starts with patented rod. "Patenting", the heat treatment before drawing, consists of a gradual heating and long soaking 300° above the critical temperature (to coarsen the austenitic grain size and to diffuse all soluble elements as uniformly as possible) followed by a quench into molten lead at about 900° F., where transformation into

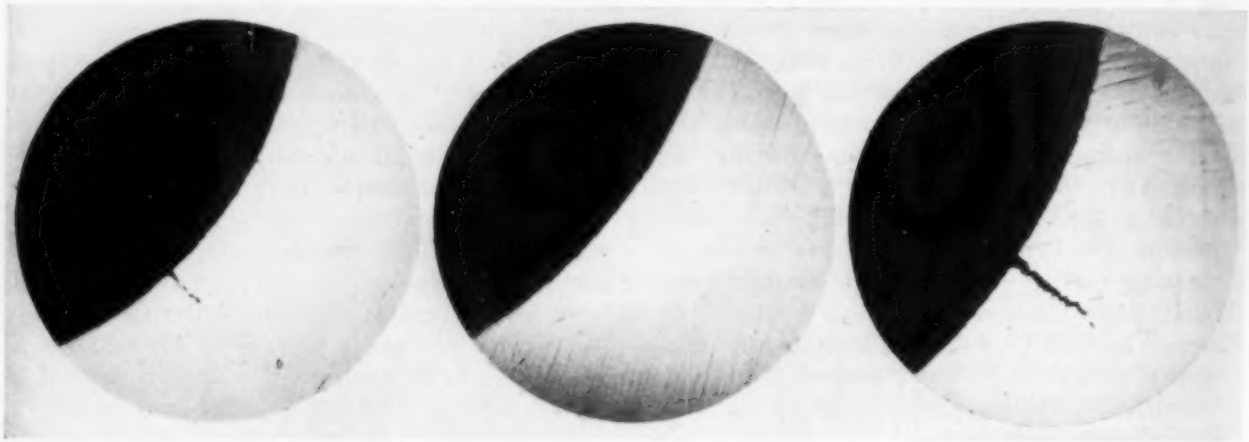


Fig. 6 — Condition of Metal on Inside of Central Loop at Point A. 40×. Left is a cracked sample of Group A, after complete processing. Center is sound metal (Group B), unaffected by caustic electrolytic cleaner. Right is a sample of Group C, cracked by a dip in hydrochloric acid

fine pearlite (called "sorbite" by spring makers) occurs quickly — complete transformation in 15 to 20 sec., according to the S-curve. This wire rod is then cleaned, baked, and drawn cold through a succession of dies to its desired diameter and ultimate strength. Its microstructure is shown in Fig. 7, and is evidently of drastically elongated grains, much distorted by slip, almost "fibrous" in nature.

On the other hand, heat treated springs or wire articles made by the "soft process", so-called, start with rods which have been given a spheroidizing anneal. A program of drafting and process anneals brings the wire down nearly to

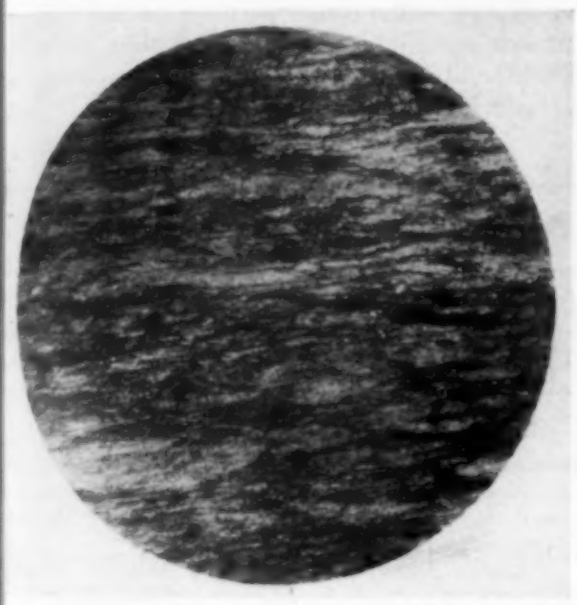


Fig. 7 — Longitudinal Section of Cold Drawn, High Carbon Steel Wire; Etched in Nital, Magnified 250 Diameters

size, and a light reduction through the last die after final anneal gives it only enough stiffness to work readily in the coiling machinery. After coiling or forming the spring is quenched and tempered. Its microstructure is shown in Fig. 8, and is tempered martensite (with excess carbide particles if the carbon content is high enough), frequently described as sorbite or troostite.

Reverting to the statement that the difference of surface condition is of greatest importance, it is at once apparent — knowing something of the manufacturing method — why this is so. The cold drawn wire has a smooth, planished surface; any dangerous surface defect existing in the original rod will be searched out and cause trouble during the drafting operations. The surface of the heat treated wire is of a different nature; unless heat treated in lead baths or in the most precisely controlled atmosphere, the surface will be slightly changed or roughened by either oxidation, carburization, or decarburization. Since springs are most highly stressed on their very surface, this difference is likely to be responsible for quite divergent performance in equivalent services. It should be apparent that slight corrosion starting at a pre-existing pit or small (almost microscopic) roughness in a heat treated spring would have a head start on the same corrosive medium attacking the truly smooth surface of a cold drawn wire.

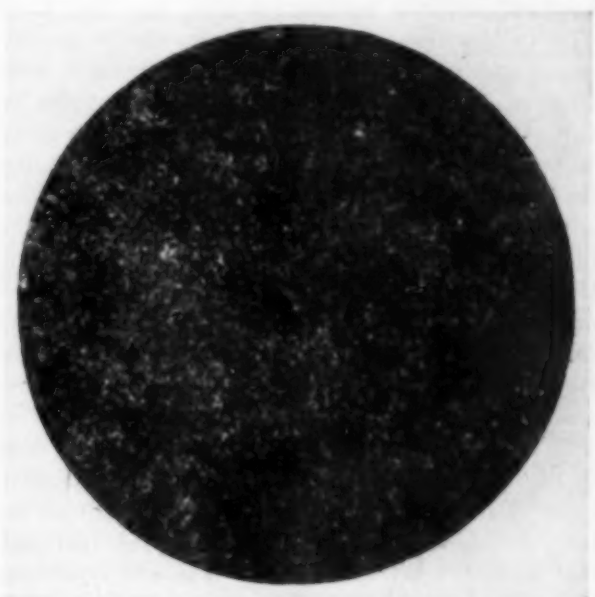


Fig. 8 — Section of Heat Treated Carbon Steel Wire, Etched in Nital, Magnified 500 Diameters

Another point is of great importance, and that is the difference in plastic action during overstrain. Even though the two classes of wire, of corresponding chemical analyses, may give almost identical results in the tensile test (averaging, for example, 225,000 psi. ultimate, 185,000 psi. "yield"★, 6.5% elongation in 4 in., and 38% reduction of area) the shapes of the stress-strain curves are notably different. The load is proportional to the elongation in the heat treated wire almost to the yield point — that is to say, the "elastic" portion of the stress-strain curve is

★Yield strength is normally taken as the stress for an agreed-upon small elongation. Such "yields" of a heat treated wire would be somewhat higher than the "yield" of a cold drawn wire of equal ultimate strength.

a straight line — and plastic action does not occur until the wire is stressed fairly close to its ultimate strength. The initial portion of an accurate stress-strain curve of cold drawn wire, in contrast, starts to curve at fairly low stresses; actual plastic action starts somewhat before the reported "yield" point, and there is a wide range of stress available for readjustment and "ironing-out" of high stresses around any existing stress-raiser before dangerous loads are approached. In other words, if atmospheric conditions leading to stress corrosion failure are present, the cold drawn wire has the better chance to survive, due to its capacity for prior internal readjustments.

Most of the last three or four paragraphs have reference to service troubles, and give little clue to the reason for the undoubtedly greater susceptibility of heat treated, high carbon steel wire to hydrogen embrittlement. It might be suggested that, since the surface layers of a piece of straight wire are in tension after quenching, these tensional forces separate the metal's atoms enough so nascent hydrogen atoms from the pickling reaction can readily penetrate and do their dirty work. However, it is equally true — according to a communication received from H. C. Boynton, consulting metallurgist of John A. Roebling's Sons Co. — that the very surface layers of a hard drawn wire are *also* in tension.

The suggestion we have most commonly met in literature and in conversation is that hydrogen embrittlement is due to the penetration of atoms into the metal along grain boundaries (through the hypothetical amorphous layers), especially those grain boundaries that are under tensile stress. For this hypothesis to have any meaning, the heat treated wire should have a lot more grain boundary areas *under tension stress* than the cold drawn wire, and they should be the locus of more intense tensile stresses.

Analyzing this situation we come up against at least two difficulties:

In the first place, is there any evidence that hydrogen diffuses more rapidly through grain boundaries than through the body of the undisturbed crystals? We have seen well documented statements in the literature that there is no such preference shown by other diffusing elements, such as carbon or oxygen.

In the second place, where are the grain boundaries in the representative micros, Fig. 7 and 8? One cannot identify anything like the well marked network shown in recrystallized cartridge brass, for instance, or annealed mild steel. It would be hard to convince yourself that there is more grain boundary in Fig. 7 than in 8.

Conclusions

Aside from this admittedly speculative digression, our brief experiments revealed corrosion cracking in the wire spring clips. From the much more comprehensive work of other researchers it may be concluded that high carbon cold drawn wire is far more resistant to stress corrosion cracking than is heat treated wire of the same strength and chemical content. Furthermore it seems evident that it is inadvisable to acid pickle heat treated high carbon steel after forming operations, or in any condition where high residual stresses are known to be present. From our brief experiments, endeavoring to stress-relieve the cold formed portions of the wire by furnace annealing, it is apparent that the range of temperature ordinarily assumed to accomplish stress relief was ineffective.

NOMINATING COMMITTEE

IN accordance with the Constitution of the American Society for Metals, President Kent R. Van Horn has selected a nominating committee from the list of candidates submitted by the Chapters. The personnel of the committee is as follows:

Walter Crafts, Union Carbide and Carbon Research Laboratories (Buffalo Chapter); Chairman.

R. M. Brick, Yale Univ. (New Haven Chapter).

C. E. Carlson, Aluminum Co. of America (Los Angeles Chapter).

Paul C. Cunnick, Rock Island Arsenal (Tri-City Chapter).

Howard E. Handy, Saco-Lowell Shops (Boston Chapter).

Walter G. Hildorf, Timken Roller Bearing Co. (Canton Chapter).

Harry K. Ihrig, Globe Laboratories (Milwaukee Chapter).

Austen J. Smith, Lunkenheimer Co. (Cincinnati Chapter).

L. C. Whitney, Copperweld Steel Co. (Pittsburgh Chapter).

This committee will meet during the third full week in the month of May, and shall name one candidate for each of the following offices:

President, for	1 year
Vice-President, for	1 year
Treasurer, for	2 years
Two trustees, for	2 years each

The committee will welcome suggestions for candidates in accordance with the Constitution, Article IX, Section 1 (b), which provides that endorsements of a local executive committee shall be confined to members of its local chapter, but individuals of a chapter may suggest to the nominating committee any candidates they would like to have in office. Endorsements may be sent in writing to the chairman or any member of the committee.

ATMOSPHERE APPLICATIONS --EQUIPMENT, METHODS AND PROCESS CYCLES

By Floyd E. Harris
Furnace Engineer
Buick Motor Division
General Motors Corp.
Flint, Mich.

A RIGID FORMULATION of the principles underlying the commercial processes for the surface treatment of steel parts has been attempted in a series of articles in recent issues of *Metal Progress*.* Numerous tests under carefully controlled conditions were described and, from data thus obtained, divers charts and formulae have been derived. Since the prime objective of these discussions has been to improve the commercial control of such atmosphere processes as carburization, decarburization, recarburization and oxide coloring, this paper will be concerned throughout with industrial practice as correlated with these derivations.

A great variety of methods may be applied to identical processing principles, and a complete coverage of all combinations of equipment and accessories, even for the process of gas carburizing, would be quite voluminous. For a proper illustration of the natural laws involved, it will be necessary to choose specific mechanisms, and these will be described only in pertinent detail. The choice of these illustrations, however, must not be construed as necessarily the ideal ones for the process; for example, the factors of simplicity and adaptability inherent in the pit type recirculating retort furnace make this unit a convenient vehicle for a wide variety of surface treatments, and for this reason it is taken for this exposition. (Likewise it is in wide use in American plants.) The chosen system of heat input control suits the peculiar needs of batch type or cyclic operations, and in general applies only to this specific type of equipment. Similarly, gas flow control, purge conditions, the generation of carrier gas and other factors are predicated by the equipment and process in question.

From the very nature of these processes, it will be difficult to divide the discussion of equipment, operating factors and actual cycles without considerable overlapping and repetition. To avoid this as far as possible, principles of equipment and operation as outlined are covered in the first section, leaving the second to deal directly with the actual processes and cycles. These are four, namely, *(a)* carburizing, *(b)* carburizing and diffusion, *(c)* replacement of surface carbon on decarburized bars and *(d)* oxide coatings. Throughout, the application of quantitative considerations will be inferred by the control choices.

*"'Case Depth' — an Attempt at a Practical Definition", August 1943, p. 265.
"Maximum Carbon in Carburized Cases", April 1944, p. 683.
"Carburizing and Diffusion Data", Data Sheet, May 1944, p. 910-B.
"An Analysis of a Typical Carburizing Gradient", June 1944, p. 1111.
"A Formulation of the Carburizing Process", September 1944, p. 488.
"Reactions Between Hot Steel and Furnace Atmospheres", January 1945, p. 84.

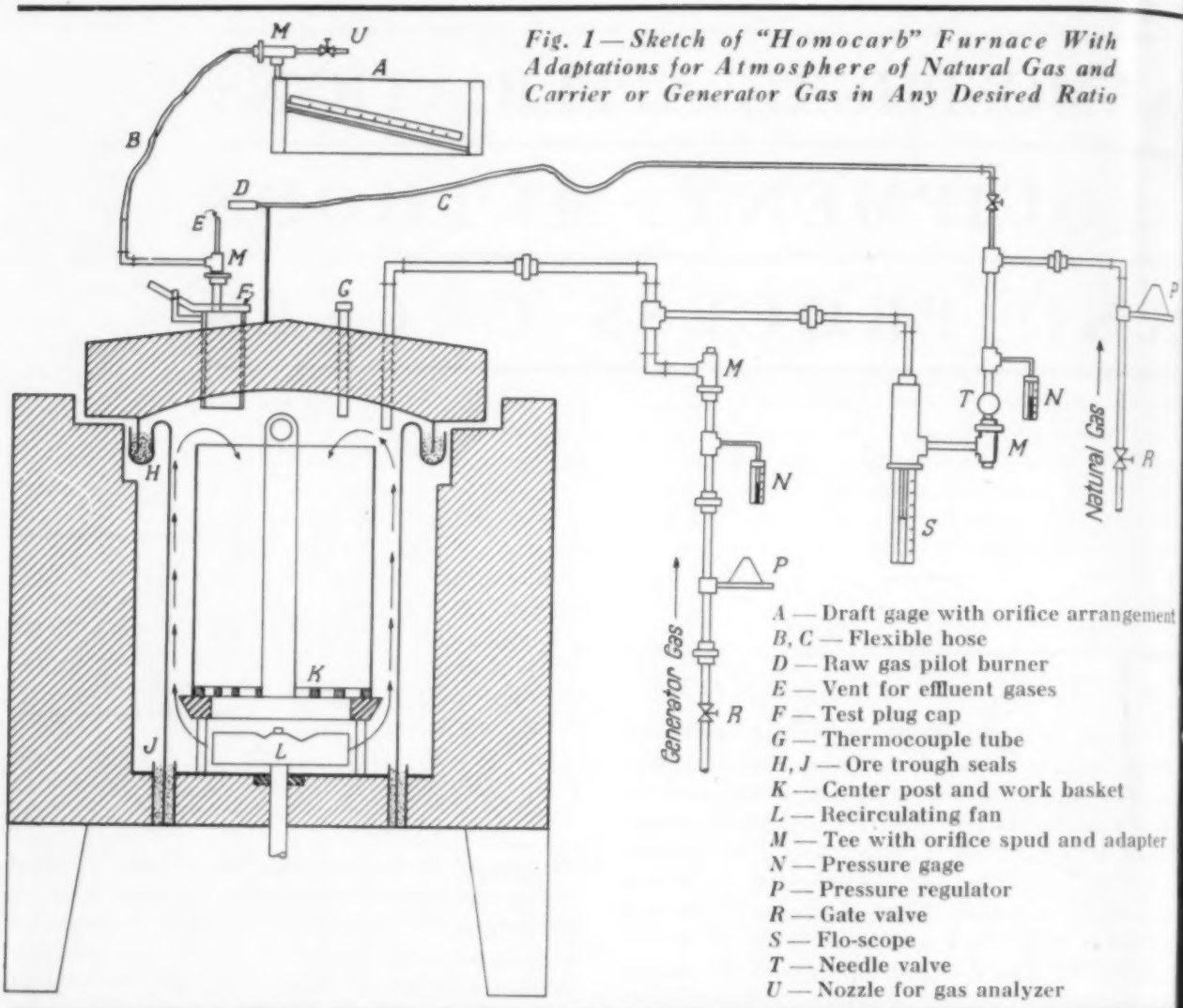


Fig. 1—Sketch of "Homocarb" Furnace With Adaptations for Atmosphere of Natural Gas and Carrier or Generator Gas in Any Desired Ratio

A — Draft gage with orifice arrangement
B, C — Flexible hose
D — Raw gas pilot burner
E — Vent for effluent gases
F — Test plug cap
G — Thermocouple tube
H, J — Ore trough seals
K — Center post and work basket
L — Recirculating fan
M — Tee with orifice spud and adapter
N — Pressure gage
P — Pressure regulator
R — Gate valve
S — Flo-scope
T — Needle valve
U — Nozzle for gas analyzer

Equipment and Methods

A conventional vertical retort, electrically heated, is shown diagrammatically in Fig. 1. This retort is sealed at the bottom with an ore trough, and a similar ore seal at the top is common to both the retort and the removable lid. These seals are capable of holding a moderate pressure (a few inches of water) without blowing. Heat is applied through elements mounted outside the retort, thus being sealed against the supplied atmosphere and being incapable of affecting its composition (and *vice versa*). A recirculating fan drives the retort atmosphere around the work basket, pulling these gases through the charge and out a perforated bottom.

Since this type of unit needs a rather dense loading in the work basket for economical operation, a differential temperature exists as the charge is being heated, the higher temperature being at the top of the charge. For this reason the thermocouple tube is located in the swinging cover, directly over the work basket. This couple and protecting tube should be readily accessible for removal, yet the construction should assure a tight seal. Because uniform and accurate temperatures are of operational importance, as well as the maintenance of retort pressures, a simple construction illustrating the position and assembly of the thermocouple tube is given in Fig. 2.

The loading characteristics of the charge determine the optimum heat input at different points of process cycles, as well as indicate the correct location of the thermocouple. A convenient rating may be made by comparing the weight of the actual charge in a given volume of symmetrical loading against the weight of a solid steel bar occupying the identical volume, and expressing the load density as this percentage weight. Thus, in Fig. 3, a load of 600 lb. per ft. of vertical height in a 24-in. diameter basket would represent a load density of 40%, since the

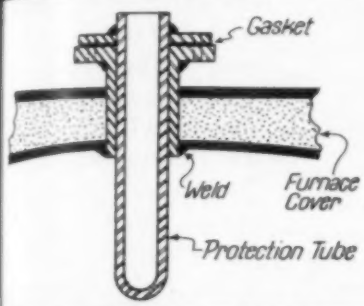


Fig. 2 — Assembly of Thermocouple Well and Protection Tube

space occupied by the charge is 3.14 cu.ft., this volume of steel weighs approximately 1500 lb., and 600÷1500=0.40 or 40%.

For close control of surface effect, a load of this density would require that the temperature, top to bottom of charge, be equalized well below the final (and critical) process temperature, and the temperature of the entire mass be brought uniformly up to final temperature. For flexibility of heat input control relative to the point-temperature measured by the thermocouple, the following method is recommended: The control-point is set on the recorder-controller at the exact process temperature, and a second contact on this recorder is set at a pre-determined temperature differential from the control-setting. This differential, below control-temperature, may be varied to suit the particular characteristics of the process and charge. Some sort of heat input controller is also necessary; it must be capable of varying the percentage of power input, and comes into operation when the registered temperature enters this differential range. This input controller may in turn be set up in various ways to match load and cycle requirements.

The above-described combination allows the operator the choice of a wide number of time-temperature-load relationships. A simple example illustrating these effects is given in Fig. 4.

Of the four heat treating processes to be discussed, three demand a carrier gas free from constituents that are oxidizing to iron, capable of removing or adding carbon to steel parts. The gas chosen for the purpose of this article is by no means the only one commercially available; however, its applicability — often with other raw gas additions — to a large number of processing requirements makes it almost an ideal carrier gas, and the elementary reactions involved in its generation afford an easy exposition of fundamental principles.

In the preparation of this carrier gas, a mixture of controlled proportions of air and natural gas is delivered to a refractory lined chamber at a moderate positive pressure by a compressor, where complete oxidation of the hydrocarbon gas forms carbon dioxide and water vapor (CO_2 and H_2O). Nitrogen (N_2) also exists as a remainder from the air. The oxygen supplied by the air is sufficient to consume all hydrocarbons such as methane. These gases leave the hot chamber, and are cooled to room temperature by passing through a water spray, thus removing the water vapor to saturation or dew point at room temperature, approximately 70° F. The gas will now be a mixture of CO_2 and N_2, with approximately 2% H_2O. It is then passed through a retort filled with hot charcoal, maintained by external heat at a temperature of 1850 to 1900° F. The nitrogen content of the gas remains unchanged, while the two completely satisfied oxides, CO_2 and H_2O, are re-formed at this high temperature by contact with the carbon (charcoal), forming the lower oxide of carbon, namely, CO, and hydrogen. This temperature (1875° F.) is sufficient for almost complete re-forming, so the final

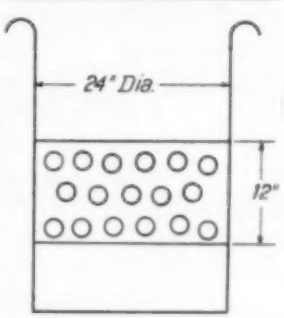
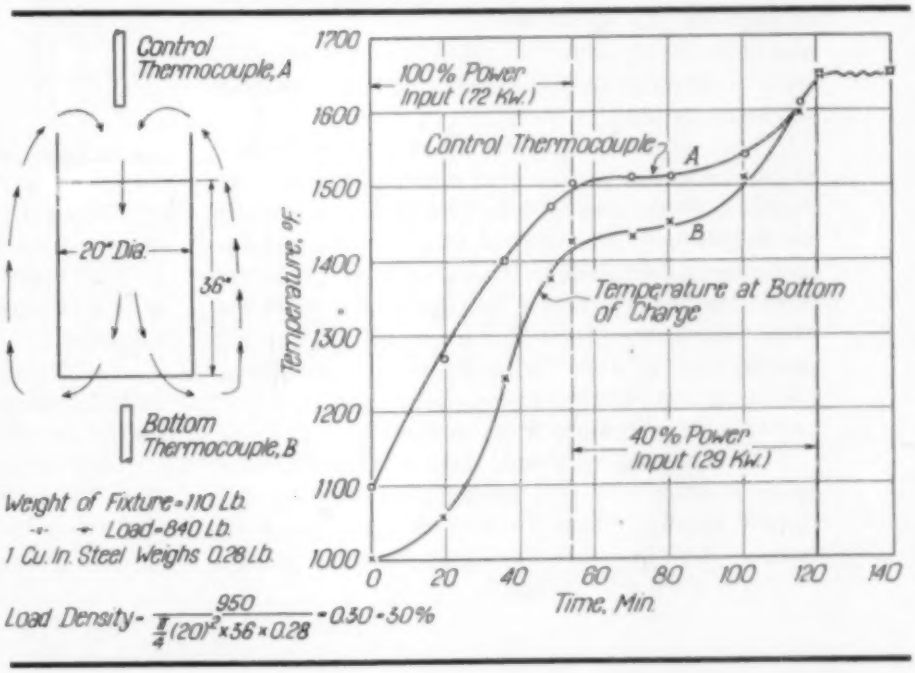


Fig. 3 — Sketch Showing Method of Computing Load Density

Fig. 4 (Below) — Sketch of Load, Thermocouple Locations and Time-Temperature Curves for Heating Cycle of Batch Type Electric Furnace



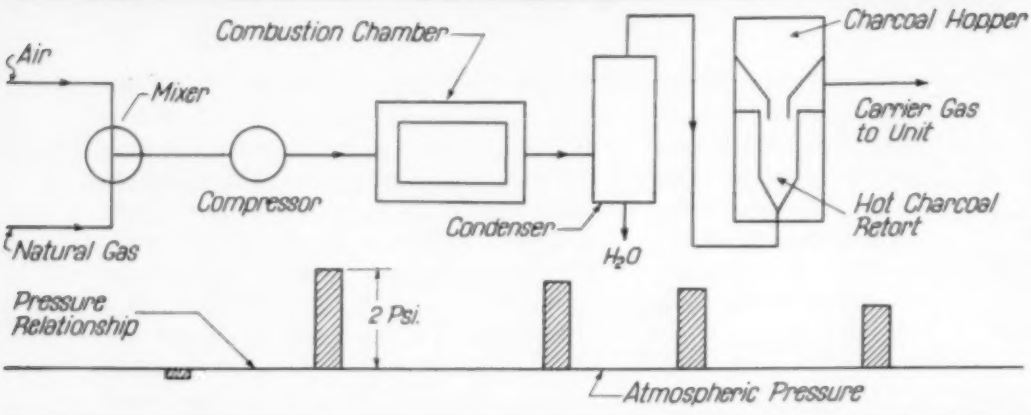


Fig. 5 — Diagram of Equipment for Generating Carrier Gas, and Relative Pressures at Strategic Locations

composition is approximately 20% CO, 2% H_2 and 78% N_2, with only enough remaining water vapor to correspond to a dew point of —20° F. or lower.

To appreciate the character of this carrier gas, it is only necessary to note that even when low percentages of CO_2 and H_2O exist in an atmosphere in contact with hot steel, these gases re-form at the expense of the carbon in the steel by the identical reaction that takes place in the charcoal retort. Similarly, the so-called pyrolitic decomposition of CO with the formation of free carbon at the elevated temperature of the carburizing reaction is extremely small, another way of saying that the carbon availability of this carrier gas is low, and there is little danger that soot will form on steel, fixtures or retort. The low hydrogen content at the same time makes the gas quite non-explosive in contact with air at relatively low temperatures, thus assuring safety in purging periods.

This generator process for the manufacture of carrier gas is represented diagrammatically in Fig. 5. Since atmosphere processing is vitally concerned with the control of gas flow, a schematic representation of the desirable pressures is shown directly below the diagram. Pressure gages at strategic points are an aid in locating sources of trouble in day-by-day operations, whenever the relationships between gas flow and pressure drop deviate from normal.

At this point, a simple device for controlling gases flowing into the work retort will be discussed briefly. This control is established by means of a pressure regulator inserted in the line ahead of an orifice spud, and the construction is such that the spud is easily removable for cleaning, and for change when the conditions require a large increase or decrease in the amount of gas to be delivered. This spud is made with a hexagonal head, fitting a standard socket wrench, and care is taken to simulate a thin plate, sharp edged orifice to obtain consistent flow coefficients. A given size of spud is limited for maximum orifice diameters, since an abnormal coefficient may be obtained if the main internal diameter of the spud is not at least five times greater than the orifice diameter. The use of such an adapter makes the spud quite accessible by removing the 1-in. pipe plug shown in the sketch, Fig. 6.

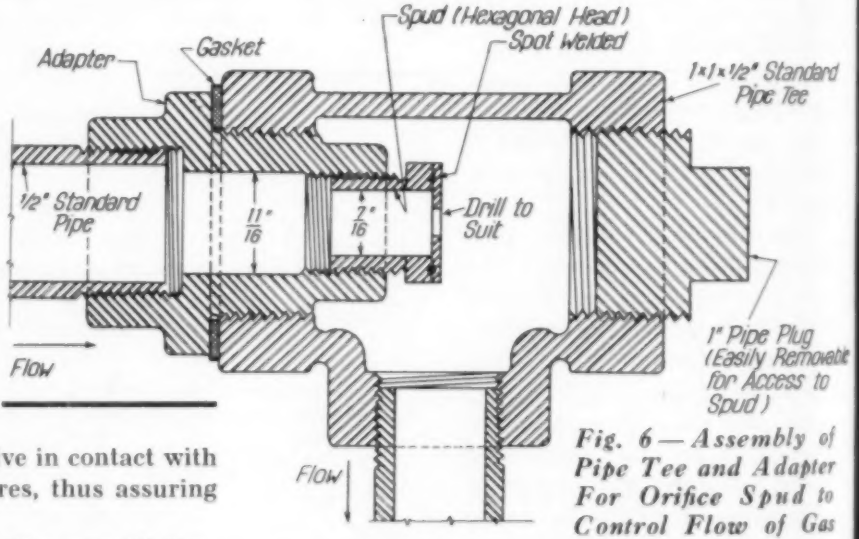


Fig. 6 — Assembly of Pipe Tee and Adapter For Orifice Spud to Control Flow of Gas

The rationale of gas supply for any reaction process may be simply stated:

1. The correct supply for the full demands of the desired reaction must be continuously supplied.

2. No gases capable of causing extraneous and detrimental reactions are to be allowed in the retort chamber.

An attempt will now be made to translate these axiomatic statements into general operating factors.

Of prime importance is the *pressure* of the

atmosphere supplied to the work chamber. This pressure must be greater than atmospheric in all parts of the retort to prevent entrainment of the surrounding atmosphere. A common error in operation is to assume that, when gases are flowing freely from the vent from the top of the retort or chamber, the latter is filled completely with the prepared atmosphere being supplied to it.

This error is caused by failure to appreciate that heated gases are much lighter than the cooler air completely surrounding the unit proper. If a pressure no greater than atmospheric is maintained at the vent, then the pressure drop at the lower portions of the retort is allowing a flow of contaminating gases through the smallest of openings that may exist. The proper sealing of the retort, as previously inferred by mentioning the ore trough and the thermocouple tube connection, cannot be neglected, and the area of the vent proper must itself be correlated to the rate of gas supply. Since the pressure at the vent is a function of the vertical height of the heated retort, a safe rule for operation is given as 0.1 in. of water column for each foot of retort height; thus a 3-ft. vertical retort should have a minimum gas pressure of 0.3 in. of water under operating conditions.

An easy check for the pressure condition should be provided. A draft gage connection as well as vent construction is shown in Fig. 1. Note that ahead of the draft gage, a very small orifice is inserted, such that the bleeder valve may be left open, allowing the operator to make an Orsat check of the gas composition without waiting for the line to purge. The pressure, of course, immediately registers on the draft gage when the pet cock is closed.

The particular rate at which gas is supplied in relation to the total working volume of the retort determines what I call a "purge out factor". Since data acquired during the purge, when properly analyzed, point directly to the operating condition of the retort, analyses of gas composition during a purge period are of value in directing the process control. Perhaps the most significant rating is the "half-life" period. This is the elapsed time, after any given moment, at which the retort gases (as indicated by their composition) contain 50% of the gas in the retort at the start and 50% of the supplied gas. In other words, the half-life period is the time required to evacuate one half of the gas existing in the retort at the beginning of the period. Assuming that no chemical reactions are taking place, the "mechanical" half-life may be readily computed for known rates of supply and retort volume, and this figure is suggested as a sound basis of judgment as to the condition of the retort, as will be explained a little later. A general solution for the half-life period, when gas is recirculated, is given in the form of a data sheet, shown at left.

The applicability of the half-life concept to a purge period may best be shown by an actual illustration. In carburizing, it is necessary that the CO_2 content be close to zero when the CO present is 20% or under; otherwise no carbon will be added at 1700° F., even if there is a hydrocarbon like methane present in the atmos-

Computation of Half-Life Period

Direct Displacement This Example is Shown for Contrast Only	*Actual, Gas Recirculated*
Out, A, In. With Direct Displacement, Change in A is Proportional to Total Supply Volume Over Retort Volume.	Out, In. With Thorough Mixing the Rate of Decrease in A is Proportional to A Itself, $dA = -A\frac{F}{V}dt$
Change in $A = -100\frac{F}{V}\cdot t$	$\int_{100}^{A}\frac{dA}{A} = -\frac{F}{V}\int_{0}^{t}dt$, Whence $\log_e\frac{100}{A} = +\frac{F}{V}\cdot t$
	For Half-Life Period, A=50
	$\log_e 2 = +\frac{F}{V}\cdot t$
	t, Half-Life $= 0.69\frac{V}{F}$

t = Time Elapsed From Zero Time in Hr.
A = Percentage of Original Volume (=100 When t=0)
F = Rate of Flow of Supplied Volume in Cu. Ft. per Hr.
V = Retort Capacity in Cu. Ft.

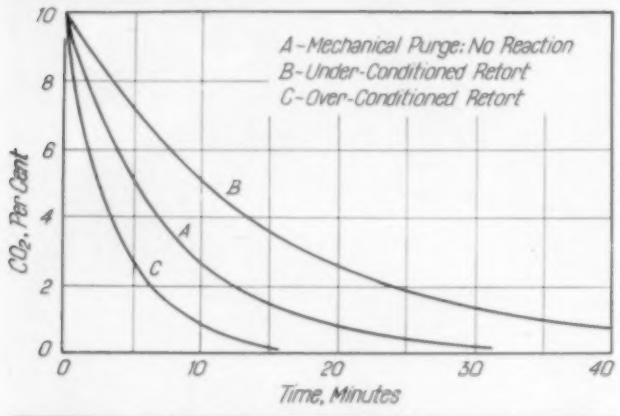


Fig. 8 — Purge Out Rate for Different Conditions of Retort — Half-Life: 5 Min.

phere. On breaking the cover seal to charge a new load at the beginning of the cycle, the retort fills with air, and on resealing and applying the customary carrier gas and natural gas supply, a quick Orsat check may indicate, say, 10% CO_2. For a half-life value of 5 min., the CO_2 check with a mechanical purge should show 5% after the first 5-min. interval, since the supplied gas contains no CO_2. Similarly the 10-min. interval would give 2.5% CO_2, the analysis after 15 min. would be 1.25% CO_2, and so on until after 30 min. the CO_2 would be reduced nearly to zero.

The illustration above assumes no chemical reaction with the retort or its contents. The actual check on the gas analyses during the purging period may vary greatly, and in either direction from the expectation for ideal conditions, and this deviation is valuable in determining the degree of "conditioning" in the work retort. To demonstrate this conditioning factor, the mechanical purge for a 5-min. half-life is indicated by the middle curve *A* of Fig. 8, representing variation of CO_2 content of the retort's atmosphere with time.

Curves of the type *B* and *C*, obtained with conditions of gas flow, load and temperature identical to the standard *A* graph, are a function of the prior history of the retort. Curve *B* indicates that the unit is "under-conditioned" for the process — for example, by an oxide coating which may easily, by reaction, increase the CO_2 percentage and prolong the purge time. Condition *C*, on the other hand, may indicate a clean working chamber, and here the reaction between methane and carbon dioxide is responsible for shortening the purge period. As may be easily imagined, such data may assist the operator's judgment materially in leading to close cycle control.

In computing the mechanical purge, care must be taken to correct gas volumes by the temperature factor. The purge formula, as given in the data sheet on page 717, must really be adjusted to temperature thus:

$$t = 0.69\ k\ \frac{V}{F}$$

where t = Half-life period in min.

F = Flow of supplied gas in cu.ft. per hr.

V = Retort volume in cu.ft.

k = Temperature factor

$$= \frac{\text{Temperature of supply gas in °F.} + 460}{\text{Temperature of retort gas in °F.} + 460}$$

Therefore the higher the temperature of the process (retort at the given time), the lower is the purge time for a given flow-volume relationship.

Transition points in some processes, where a change in atmosphere composition is required at some particular level of temperature, or after some particular elapsed time, may also depend on the purge factors for an accurate control of the transition interval. Examples of this condition, as well as of the other common operating factors, may be noted in the following section on process cycles.

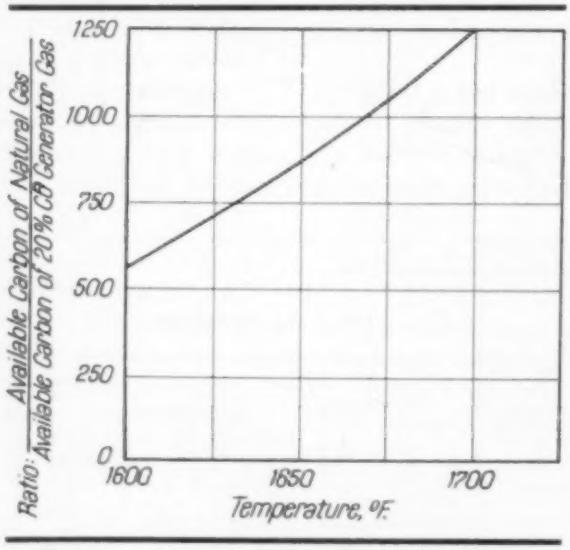


Fig. 9 — Methane Has Over 1000 Times as Much Available Carbon as the Carrier Gas in the Carburizing Range. "Available Carbon" is defined in the article for January 1945, p. 88

Process Cycles

Applications of the general control principles briefly outlined will now be described for a few specific atmosphere processes. By employing the same type of heating equipment and identical principles in the control methods, the demands of individual process requirements should illustrate by contrast the role of gas reactions in obtaining desired surface effects on steel parts.

Shop Process Sheet

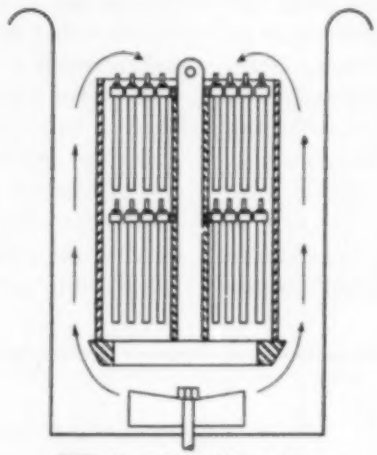
OPERATION: CARBURIZING ACCESSORY DRIVE SHAFT

Steel: S.A.E. 4620; Case: 0.040 to 0.044 in. Weight of part: 6 lb.

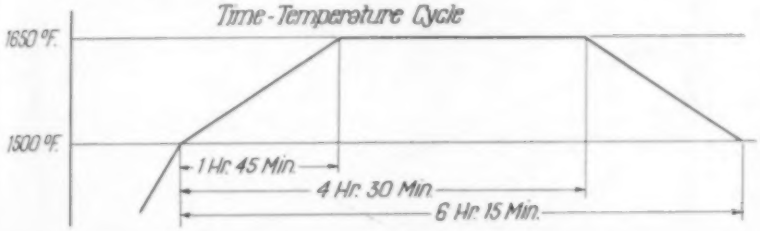
LOAD (MAX.)	LB.
2 Trays (M-2709) at 35 lb.	70
2 Spacers (M-2951) at 20 lb.	40
1 Base & Post (M-2952)	138
1 Shell (M-2953)	110
Fixture weight:	358
Shafts — 2 layers at 70 pieces or 140 at 6 lb.	840
Total weight (fixture + load)	1198

OPERATION DATA

1. Recorder at 1650° F.; Controller operating from 1500°F. to 1650° F.
2. 50 cu.ft. per hr. Drycolene; Minimum furnace pressure 0.30 in. H_2O.



☛ Load Uniformly



OPERATION	TIME, HOURS	°F.	NAT. GAS*	% CO_2	CHECK PLUG CASE	POWER INPUT
Charge	0	—	10 to 13	—	—	40%
Check CO_2	1.25 to 3.00	1500 to 1650	10 to 13	0.0	—	40%
1st test	5.75	1650	0	—	0.034 in.	0
Pull	7.50	1500	0	—	0.040 to 0.044	0

*Cu.ft. per hr.

NOTES: CO_2 must be 0.0% before carburizing starts. If CO_2 is high, continue checks and note time when CO_2 is less than 0.2%.

Shut off control switch and leave fan on when test plug shows 0.034 in. case. (This marks beginning of diffusion cycle.)

Pull test plug at 1500° F.; it should have 0.040 in. case or more.

Carburizing — The supply of carbon to the steel surface from a gaseous system parallels closely its ability to decompose, as demonstrated by the familiar formation of soot. In other words, a carbon-bearing gas that soots readily in the carburizing temperature range will as readily furnish carbon to the steel surface, while a stable gas will furnish carbon for neither reaction — it will neither soot nor carburize. The problem is one of supplying sufficient carbon to satisfy the steel surface exposed, since the steel demand is a limiting factor, and to also restrict any excess supply of gas to the degree that no great amount of free carbon can be deposited. At the same time, conditions of pressure and purge-out, as already indicated, must be met if detrimental reactions are to be avoided. In order to control these conditions and reactions, a dual gas is indicated — that is to say, a lean carrier gas plus a carbonaceous gas.

The function of the carrier gas, then, is not to supply carbon. Rather, a gas incapable of sooting in the carburizing range, and containing no oxidizing or decarburizing components, can be used to best advantage in maintaining adequate pressure and purge requirements, since the supplying of carbon to the steel and its rate of flow have no connection. Therefore the flow of this gas can be maintained at a constant rate, and this optimum rate will be a function of the equipment demands, rather than of the requirements of the carburizing cycle.

The "carbon supply" medium, natural gas, in contrast, will be supplied at a variable rate, since the demand for carbon will be a function of the "prior history" of both the retort chamber, in its conditioning, and the work load.

A comparison of these two gases is in order.

From the reaction standpoint the carrier gas will be taken simply as a mixture of gases whose CO content is 20%, while the reactive supply gas will be considered as pure methane, CH_4, since that is the principal constituent of natural gas. On this basis, a comparison is shown in Fig. 9, of the relative strength of these two media in the normal carburizing range, 1600 to 1700° F. From this curve it appears that at 1650° F. the available carbon for 1 cu.ft. of natural gas is equal to that of 800 cu.ft. of carrier gas. The relative strength of these two supply media, in terms of a normal flow of carrier gas to the retort furnace described earlier (say 50 cu.ft. per hr.), indicates that a supply of 50÷800 or 0.06 cu.ft. per hr. of natural gas flow

would supply as much carbon to the steel as the carrier gas. From this consideration, it may be surmised that the ideal carrier gas should contain no trace of methane.

A shop process sheet is shown on page 719 which includes applications of these principles as applied to a typical production operation. Here, a 6-lb. shaft, protected in part with copper plate, is to be carburized to a depth of 0.040 to 0.044 in., and these shafts must be slowly cooled after carburizing, without loss of surface carbon. No metal-to-metal contact is allowable, so 140 of these shafts are suspended vertically on suitable fixtures so that the surfaces to be carburized are exposed to the atmosphere gases. The charge includes 840 lb. of stock, and 358 lb. of fixture and container, a total of 1198 lb. Since the container is 20 in. round by 36 in. high, the load density is 30%, a good value for this type of process.

A standard test plug, ⅜ in. round, of S.A.E. 1020, is removed through the test plug cap at the time noted, held until the temperature reaches approximately 1450° F., then quenched in water. This bar is fractured, ground, and given a 10-sec. etch in a 50% nitric acid solution, and the total darkened area is read with a Brinell glass. This reading checks well with case depth as defined in previous articles in this series. This check takes about 4 min. total time.

Immediately after charging and sealing the cover carefully, the power is turned on (72 kw.), as well as the carrier gas (50 cu.ft. per hr.) and the natural gas (12 cu.ft. per hr.). To protect the copper plate from oxidation, the CO_2 content in the retort atmosphere must be reduced quickly before even a moderate temperature is reached. The purge factor, with a half-life period of less than 5 min., attends to this. The temperature recorder setting is 1650° F. and at 1500° F.—150° F. below the control point — the input controller automatically reduces the power to the controller setting, in this case 40% or 29 kw. Thus, the entire charge reaches a uniform temperature before the carburizing rate is important.

Before the charge reaches the control point, 1650° F., an Orsat gas analysis for CO_2 should be made, and this reading should approach zero. Since a zero CO_2 reading with an ample supply of CH_4 insures normal carburizing, this check is a safety precaution on operational procedure. A test plug is removed 5 hr. and 45 min. after charging, quenched and broken to check the case. It is found to be 0.006 in. less than the required case; the power switch is then pulled and the natural gas flow is stopped, allowing a slow cool of 2 hr. to reach 1500° F., with the carrier gas on.

This slight diffusion treatment insures against high carbon content at the surface (to

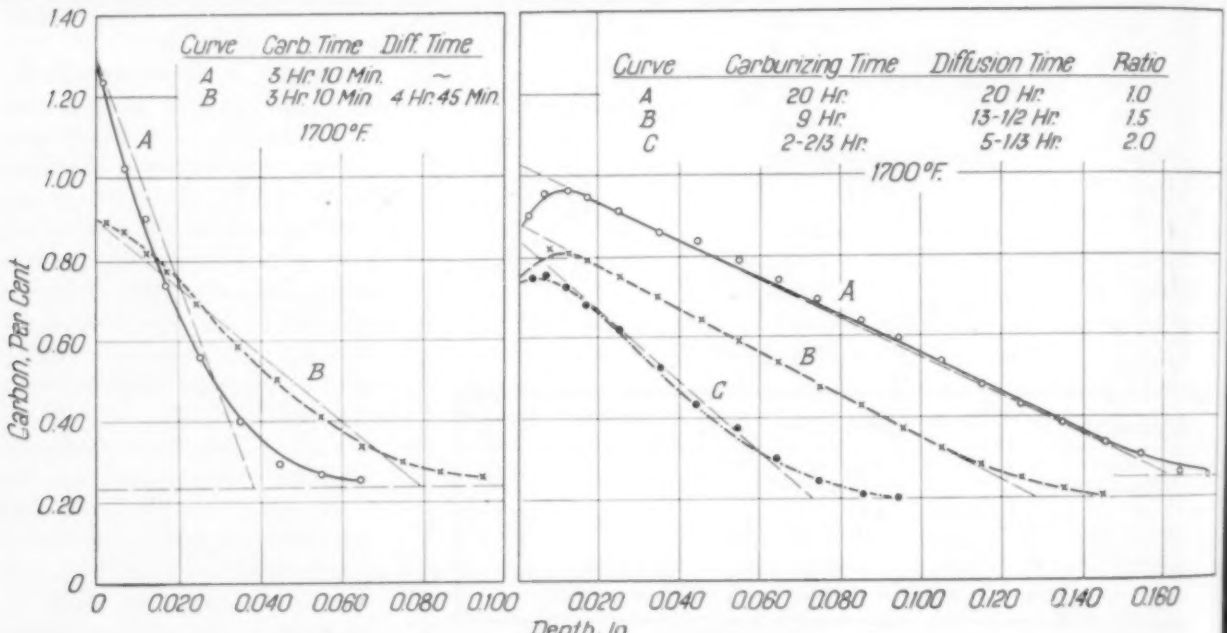


Fig. 10 (Left) — Gradients for a Carburizing Run Followed by a Somewhat Longer Diffusion Time
Fig. 11 — Carbon-Penetration Gradients for Various Ratios of Diffusion Vs. Carburizing Times

Log of Carburizing-Diffusion Runs (Fig. 12)
Constant flow of 55 cu.ft. per hr. of generator gas

Time	Temperature	% CO_2	Natural Gas*	Steam†	Remarks
		Decarburization of Bars *A*, *B*, & *C*			
11:30	—	—	0	4.5	Steam on
1:15	—	3.9	0	4.5	Bars in
1:30	1650	4.7	0	3.0	
2:00	1650	4.5	0	2.0	
2:30	1650	4.5	0	0	Steam off
2:45	1650	2.2	16	0	Bars *A* and *C* out; natural gas on
		Recarburization of Bar *B*			
3:00	—	0.1	16	0	
3:15	1650	0.0	16	0	
3:30	1650	0.0	10	0	
3:45	1650	0.0	0	0	Natural gas off
4:30	1650	0.0	0	0	
5:45	1650	—	0	0	Bar *B* out
		Recarburizing Bar *C* along with 9 similarly decarburized plates, 0.40 x 25 x 8 in.			
2:50	—	—	12	0	Stock in
2:55	1460	10.4	12	0	
3:00	1470	2.2	12	0	
3:05	1490	0.8	12	0	
3:10	1530	0.3	12	0	
3:15	1560	0.1	12	0	
3:20	1600	0.0	12	0	
3:35	1650	—	12	0	
4:00	1650	0.0	0	0	Natural gas off
4:20	1650	0.0	0	0	
4:45	1650	0.0	0	0	
5:10	1650	0.0	0	0	
5:30	1650	0.1	0	0	Temperature controller set at 1625° F.
6:00	1625	0.0	0	0	
6:15	1625	0.0	0	0	Stock out

*Cu.ft. per hr. †Pressure, psi.

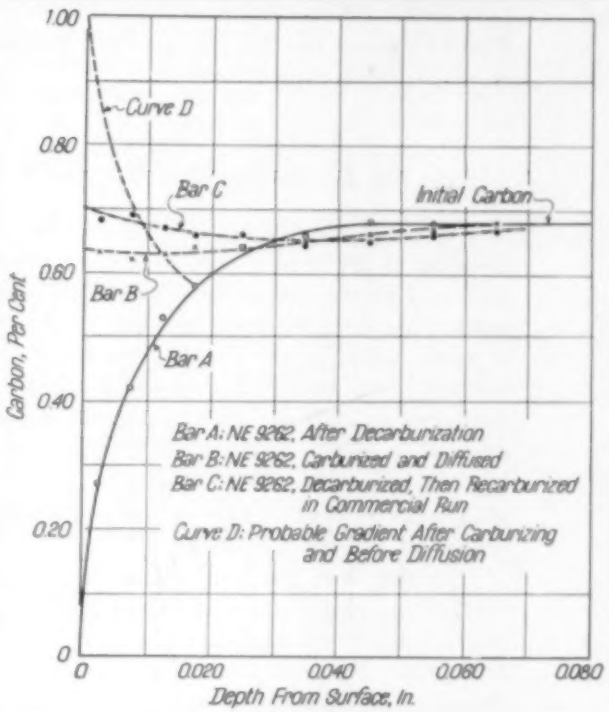


Fig. 12 — Carbon-Penetration Gradients After Test Runs and a Commercial Run to Restore Carbon to Decarburized Plate of NE9262 Steel

minimize grinding checks) while the low temperature of 1500° F. at discharge allows the work to be slowly cooled without danger of undue surface decarburization. It will be noted that this cycle conforms to the case depth chart (Table V of the article in August 1943, p. 271), and this procedure will obtain uniformity of cycle times, as well as of product, throughout the charge.

Carburizing and Diffusion

When the diffusion time of the process is of longer duration than the time of carburizing, the initial conditioning of the retort should be carefully checked before attempting the process. For the straight carburizing process, with a moderate diffusion cycle as noted above, an over-conditioned unit may be safely employed, since the surface carbon concentration is maintained relatively high. As the ratio of diffusion to carburizing increases, with the idea of lowering the surface concentration and of obtaining, at the same time, exact gradients of a predictable nature, care must be taken that the absorption of methane in soot deposits does not carry over into the diffusion period. Periodic Orsat analyses for CO_2 gas during the purging period (during which time a moderate amount of natural gas is added to the carrier gas) should follow closely the form of the mechanical purge shown as curve *A* in Fig. 8. Care is also taken, during the carburizing period, to throttle the flow of natural gas as the demand for carbon lowers, in order to avoid an over-conditioning effect during this period.

Figure 10 shows the gradient for a carbon concentration bar after the carburizing period as well as the final gradient form. In Fig. 11 is shown a group of final gradients, for different times of carburizing from long to short and various ratios of diffusion to carburizing times. These examples are to illustrate the greater care necessary when consistent modifications from a normal gradient are desired. Throughout, the consistency of case depth-time results obtained

will be noted. Regardless of diffusion time, *total* case depth follows the case depth chart (given in *Metal Progress* for last August) for the *total* time of carburizing plus diffusion.

Carbon Recovery on Decarburized Surfaces

A good illustration of the carbon recovery problem is given in a test on a group of NE9262 bars and plates, where in consecutive periods the pieces are decarburized without scaling, carbon is next added with a steep gradient from the surface, and lastly this gradient is leveled by diffusion to conform to the correct carbon concentration of the heat of steel. These graphs of Fig. 12, page 721, are obtained by removing and studying consecutive bars at the end of each cycle or period of heat treatment.

In the first portion, steam is added to the carrier gas to maintain the decarburizing atmosphere, and the adaptability of this carrier gas will be further demonstrated if it is noted that when steam is replaced by methane the CO_2 drops from 2.2% (a decarburizing atmosphere) to 0.1% (a carburizing atmosphere) in a period of 15 min., as shown in the log sheet at left of Fig. 12.

The results of the test runs are shown by curves *A* and *B*. Figure 12 also shows (curve *C*) the results of a commercial run in which a decarburized bar from the test run is included with a load of NE9260 stock that had a decarburized surface very similar in character to the test bar. It is seen that the commercial run checks quite closely with the test run (except of course that no preliminary decarburizing period is maintained) both in the log sheets and the final carbon gradients obtained.

In such corrective heat treatments the retort atmosphere must be closely checked during the individual purge period after loading, if time and rate of natural gas supply are to be properly estimated for the carbon addition period. This is particularly true when the initial carbon concentration at the surface of the decarburized bars is low, and when the depth of decarburization is relatively great. This is shown on a recovery run for "Nitralloy G", where the thickness of the decarburized zone is about 0.070 in. (See Fig. 13.) Here, by throttling the natural gas supply during a portion of the purge period, a condition simulating somewhat the test bar run previously shown in Fig. 12 is attained, since the CO_2 is decreased slowly during this early part of the run. This insures, to a degree, that the retort is not overconditioned, and allows a closer control of the carbon added.

Carbon Restoration in Nitralloy G Bars (Fig. 13)
Constant flow of 90 cu.ft. per hr. of carrier gas

Time	Temp.	% CO_2	Natural Gas★	Remarks
1:55 P. M.	—	—	6	Stock in
2:00	1510	10.2	6	
2:05	1600	5.7	4	
2:10	1630	4.9	4	
2:15	1650	4.5	6	
2:25	1650	2.8	6	
2:35	1650	0.9	6	
2:45	1650	0.4	6	
2:55	1650	0.1	6	
3:00	1650	0.0	6	
3:15	1650	0.0	0	Natural gas off
4:15	1650	0.3	0	Temperature control set at 1700° F.
8:15 A. M.	1650	0.4	0	
8:30	1730	0.2	0	
8:45	1700	0.3	0	
9:00	1700	—	—	Stock out

★Cu.ft. per hr. of natural gas.

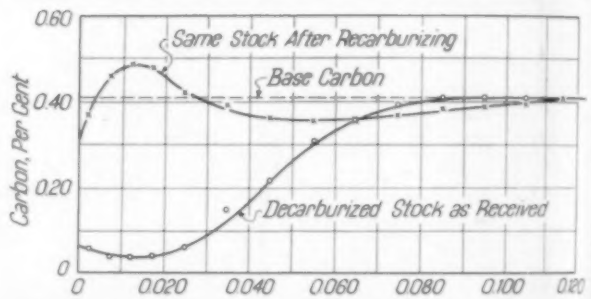


Fig. 13 — Carbon-Penetration Gradients of Decarburized Nitralloy Steel as Received and After Corrective Heat Treatment

Oxide Coatings

The production of a tight black oxide coating on cast iron or steel parts at temperatures under 1150° F. provides a good illustration of the dual possibility of analyzing the chemical reactions between gas and steel surface, and controlling the necessary heat treatment cycle. This oxidizing operation will be described, briefly.

The desired oxides are FeO and Fe_3O_4, and the problem is to prevent the formation of Fe_2O_3, or red rust. The rusting of bright steel parts at room temperature is a familiar reaction, in which water (in the liquid form) on the steel surface reacts, in contact with air, with iron to form ferric hydroxide, which decomposes according to the following chemical reaction to form the undesirable oxide:

$$2Fe(OH)_3 = Fe_2O_3 + 3H_2O \qquad (1)$$

Not so well appreciated is a reaction in the vapor phase, written as

$$H_2O = OH + \tfrac{1}{2}H_2 \qquad (2)$$

which competes with the common reaction showing the decomposition of water vapor as the temperature rises:

$$H_2O = H_2 + \tfrac{1}{2}O_2 \qquad (3)$$

Reaction (3) is much faster than reaction (2), and at temperatures above about 900° F. the oxide Fe_3O_4 (which is the stable oxide up to 1050° F.) is readily formed, providing no air is present in the steam atmosphere to furnish some oxygen, O_2. It will be noted that no appreciable amount of O_2 is available above the amount necessary for the formation of oxide — that is, the O_2 in the gaseous form as found in the retort and resulting from the decomposition of steam is always extremely small. Above a temperature of 800° F. reaction (2), or decomposition to the hydroxyl radical, possesses sufficient energy so that, with an appreciable O_2 content, a chain reaction with free (uncoated) iron promotes the formation of ferric hydroxide, which breaks down to ferric oxide as in reaction (1). This reaction is very fast. Once the black oxide has been formed so that the free iron surface is not exposed, this reaction no longer can go forward, and an excess supply of oxygen does no harm to the coating, nor will it change the process rate.

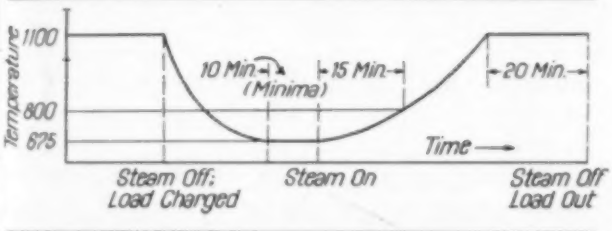


Fig. 14 — Time-Temperature Graph of the Ferrox Process

The demands of the heating cycle during the so-called Ferrox process are, therefore:

Temperature Range of Stock	Atmosphere
70° to 212° F.	*Dry air*, no H_2O
212° to 800° F.	Any mixture of air and steam for any time period
800° to 1100° F.	H_2O vapor, *no air*
Cooling to room temperature	Air; the coating is the protection

The operating cycle in production follows: The recorder controller is set at the control temperature, 1100° F. The input controller range, beginning 425° F. below the control point setting, is 675 to 1100° F. Before removing the processed load, the input controller (automatic in this case) is set at zero power input, and the controller switch is turned to the "Off" position, discontinuing the automatic feature which controls the input setting. Steam flow is also interrupted.

On removing the hot load, the fresh charge (with parts free from water) is inserted, and the cover replaced but only lightly sealed. The heavy charge lowers the retort temperature until the thermocouple reaches 675° F., and this temperature indication is held for 10 min. to insure that all parts of the load are well above 212° F. The steam flow (half-life under 2 min.) is then started, the cover is now *tightly* sealed, and the power input controller switched on. The input increases slowly, so that 15 min. have elapsed before any part of the load may reach 800° F. By this time the retort is completely purged of air by the steam flow. Hold for 20 min. after reaching 1100° F. and the cycle is completed. Steam is then turned off. The time-temperature graph, Fig. 14, is illustrative.

Summary

This is the fifth and last in a series of articles dealing principally with the supplying of carbon to steel surfaces. The first four are concerned with quantitative deductions featuring the demand characteristics of the product, and the supply properties of gaseous media. The present paper suggests the manner of correlating this supply and demand under conditions of commercial practice.

The concentration-depth graphs, shown frequently throughout, have been obtained with production equipment and facilities. All tests concerned with fundamental data, illustrated in the first three of the series, were made under conditions of load and operation chosen to insure quite exact reproducibility; those in this latest presentation of commercial applications are of course examples of commercial control.

A few references are given where the data or methods noted are used directly in the text. In general, the attempt has been to interpret the data rather than to review the literature. Perhaps, for contrast, more examples of reactions which interfere with the direct or quantitative control of concentration gradients would have been illuminating. For example, the oxide reaction at low temperatures as influenced by excessive quantities of free oxygen (described immediately above) affords a visual proof of extraneous influence, and this same hydroxide reaction often explains abnormal results in the practice of carburizing.

A PROPOSED MATCHED SET OF STEELS

By J. E. Erb
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General Electric Co.
Schenectady, N. Y.

"WE ARE IN the predicament that our facts have outstripped our knowledge and are now encumbering its march. The publications of our scientific institutions overflow with minute and countless details which perplex the judgment and which no memory can retain. In vain do we demand that they should be generalized and reduced into order. Instead of that, the heap continues to swell. We want ideas and we get more facts. We hear constantly of what Nature is doing, but we rarely hear what man is thinking. Owing to the indefatigable industry of this and the preceding century, we are in possession of a huge and incoherent mass of observations which have been stored up with great care, but which, until they are connected by some presiding idea, will be utterly useless."

This could well be the plaint of a materials engineer, today laboring to find the best material to put into a given machine part. As a matter of fact, it is a quotation from Buckle's "History of Civilization in England", written about 1850.

A large proportion of the needs of steel consumers could be satisfied with no more than sixteen different compositions, as proposed in the large table on page 726. This includes not only the common structural and machinery grades, but also the so-called toolsteels and stainless.

An article on "Steel Simplification" recently compiled and published by G. W. Birdsall in *Steel* for April 24, 1944, explains the urgent need for simplification, and indicates the benefits which would result. F. R. Palmer of Carpenter Steel Co., who has done so much to clarify the toolsteel situation, has (perhaps without intending to) provided the presiding idea for the table now to be presented.

In the opinion of the writer, simplification of the proper sort would result not only in enormous economies for American industry but also in improved quality of product. However, it is not the purpose of this article to enlarge upon the need for simplification nor its benefits, but rather to set forth a concrete, definite proposition in the hope of provoking discussion and — more important — concerted action by makers and users for the benefit of all.

Therefore it seems desirable, first of all, to establish the background of this proposal by sketching briefly the natural evolution of the present situation, as we see it.

Development of Alloy Steels — Fifty years or more ago practically all steels were of the plain (non-alloy) variety, graded according to carbon content or "temper". These served their purpose very well, and, in many respects, still do. They are represented in the large table by the first vertical column on the left.

The increase in size and power of machinery made it necessary to obtain properties in large sections beyond the capacity of the plain carbon steels. In the opposite direction, the demand for higher speeds and lighter weight called for properties which the carbon steels could easily have met had it been possible to water-quench them without excessive rejections of parts cracked or warped out of shape. Hence the demand for oil hardening or air hardening steels. Out of all this

grew the vast conglomeration of alloys we have today. The legitimate purpose of the steels of Column II can therefore be summed up in two words — hardenability and quality.

The meaning and control of *hardenability* have gradually come to be understood and applied. However, the interpretation and importance of *quality* are not so well founded. This has operated to retard standardization — a point which will be discussed later on.

From another direction came the demand for better manufacturing characteristics, such as better machinability or better ductility (drawability). This gave rise to the free cutting steels and the deep drawing steels, which are lumped together in Column III.

Another improvement was in the direction of longer life — that is, resistance to abrasion and corrosion, both of which are closely related. High speed toolsteel is, in effect, a long-lived steel, since it will last longer than carbon steel when cutting at the same speed, or work faster for equal life. Cemented carbides and the various cast tool alloys and hard-facing materials may also be considered to be in the category of Column IV.

Column V comprises the steels which truly resist corrosion, and might be extended to include the heat resisting steels and alloys, since they are very much the same.

So we have at a glance the general steel situation which, to the writer at least, presents its true perspective.

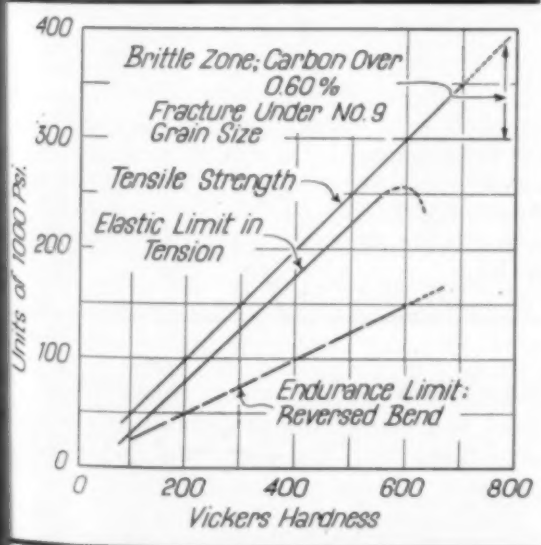


Fig. 2 — Relation Between Hardness and Tensile Strength, Elastic Limit and Endurance Limit of Carbon and Alloy Steels

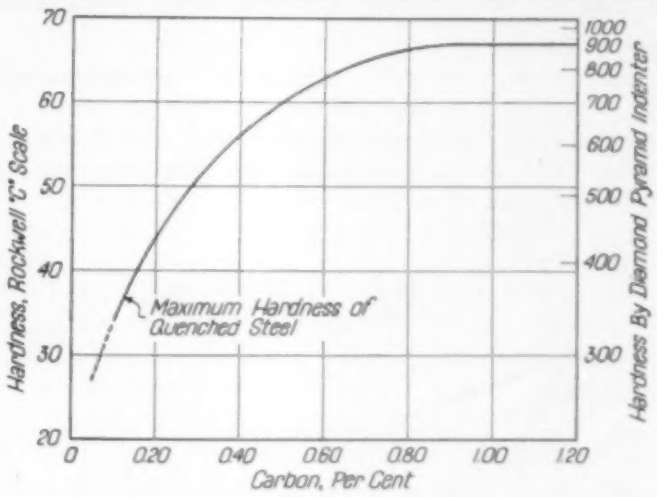


Fig. 1 — Relation of Carbon in Plain or Alloy Steel to the Maximum Hardness Attainable by Quenching

Fundamental Relationships

Alloy Vs. Carbon — The commercial separation of steels into "alloy" and "carbon" has done much to confuse the fundamental issue. The alloys extend and supplement the carbon steels in a continuous manner, as shown by the large table on page 726. Therefore, there should be no sharp separation between them, for carbon is, after all, the predominant alloying element in steel. More than any other, it controls hardness, strength, and other properties. First, the maximum (potential) hardness obtainable by quenching depends solely upon carbon and not upon alloy content (see Fig. 1). Next, the tensile strength, fatigue strength, and even the elastic limits are largely functions of hardness — and therefore carbon content — as depicted by Fig. 2.

The latter idea was advanced, with ample supporting evidence, over 20 years ago by E. J. Janitzky, and is elaborated in the 1939 edition of Metals Handbook, pages 515 to 518. Some readers will contend that the relationships shown by this diagram are only approximate, but how often in practice does the engineer know the actual stresses with greater precision?

So we can say that carbon occupies the primary, and the other alloys a secondary, or supplementary, place in the general map of steels.

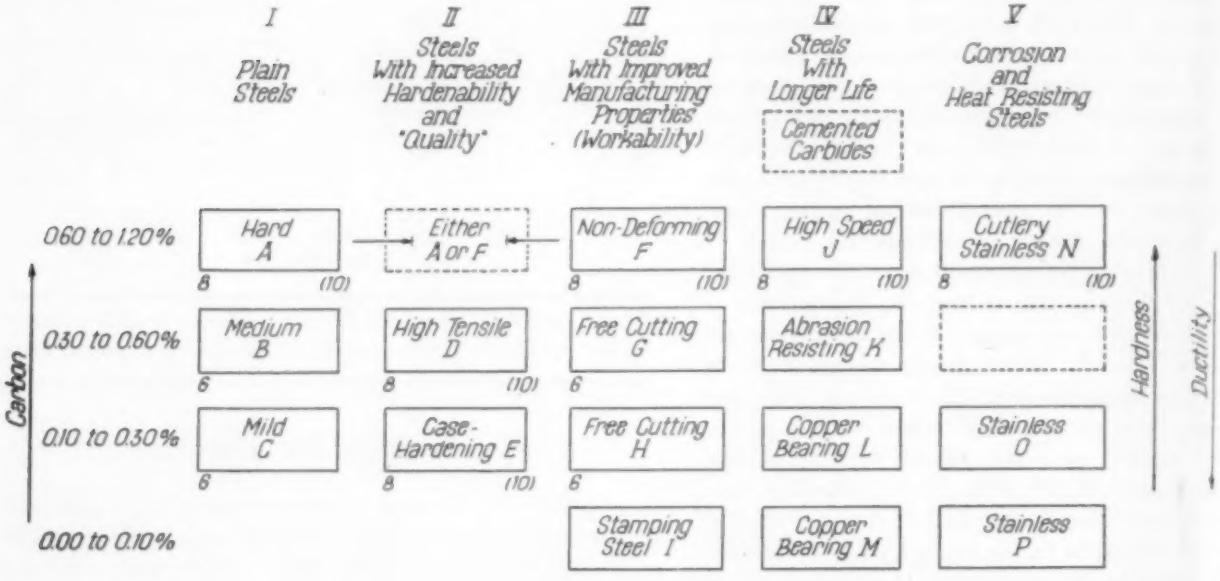
Quality; Cleanliness and Fine Grain — One of the main causes of the present situation is that users have, for one reason or another, turned to the use of panaceas in the form of new alloy combinations instead of seeking that particular and definite *quality* which is what they really need.

Disregarding the more obvious defects such as

A Proposed "Matched Set" of Steels

By J. E. Erb, with acknouledgments to Frank R. Palmer

Figures represent minimum fracture grain size; if in parentheses, the desired grain size; otherwise fracture grain size is not specified.



Steel	Carbon	Manganese	Silicon	Sulphur	Phosphorus	Chromium	Nickel	Molybdenum	Vanadium	Others
A	1.02/1.08	0.20/0.40	0.20/0.40	0.025 max.	0.020 max.	0.10 max.	0.10 max.	0.10 max.	0.20/0.30	—
B	0.47/0.53	0.60/0.90	0.15 min.	0.045 max.	0.035 max.	—	—	—	—	—
C	0.22/0.28	0.60/0.90	0.15 min.	0.050 max.	0.040 max.	—	—	—	—	—
D	0.52/0.58	0.60/0.90	0.40/0.60	0.025 max.	0.020 max.	0.90/1.15	0.40/0.60	0.20/0.30	0.20/0.30	—
E	0.14/0.20	0.60/0.90	0.40/0.60	0.025 max.	0.020 max.	0.90/1.15	0.40/0.60	0.20/0.30	0.20/0.30	—
F	1.02/1.08	0.60/0.90	0.40/0.60	0.025 max.	0.020 max.	1.40/1.60	0.40/0.60	0.20/0.30	0.20/0.30	—
G	0.47/0.53	1.30/1.60	0.15 min.	0.10/0.20	0.045 max.	—	—	—	—	—
H	0.14/0.20	1.30/1.60	0.15 min.	0.10/0.20	0.045 max.	—	—	—	—	—
I	0.07/0.13	0.30/0.60	0.15/0.45	0.050 max.	0.040 max.	—	—	—	—	—
J	0.80/0.86	0.20/0.40	0.20/0.40	0.025 max.	0.020 max.	3.80/4.20	—	4.80/5.20	1.40/1.60	4.80/5.20 W
K	0.47/0.53	0.60/0.90	1.40/1.60	0.050 max.	0.040 max.	0.20/0.30	0.40/0.60	—	—	0.20/0.30 Cu
L	0.22/0.28	0.60/0.90	1.40/1.60	0.050 max.	0.040 max.	0.20/0.30	0.40/0.60	—	—	0.20/0.30 Cu
M	0.07/0.13	0.30/0.60	0.15/0.45	0.050 max.	0.040 max.	0.20/0.30	0.40/0.60	—	—	0.20/0.30 Cu
N	0.80/0.86	0.60/0.90	0.40/0.60	0.025 max.	0.020 max.	16.0/18.0	1.40/1.60	—	0.90/1.10	4.80/5.20 Co
O	0.09/0.15	0.60/0.90	0.40/0.60	0.025 max.	0.020 max.	11.5/13.5	—	—	—	—
P	0.09/0.15	0.60/0.90	0.40/0.60	0.025 max.	0.020 max.	18.0/20.0	8.0/10.0	—	—	—

pipe, seams, and thermal cracks, cleanliness and grain size are the most important characteristics in those steels which are to be hardened or which are to take a high finish.

In regard to size and character of inclusions, there seems to be fairly general (but not coordinated) agreement as to what can be tolerated and what cannot — although much still remains to be learned in this respect. At any rate, we believe it will be pretty generally conceded that harmful inclusions will be no more characteristic or inherent in steels of the proposed analyses than of any others, and, therefore, this factor should not stand in the way of simplification.

With regard to grain size, the issue is more cleanly cut:

The importance of a fine fracture is an old story to heat treaters and those engaged in the manufacture of tools and hardened parts in general. This is especially emphasized by F. R.

Palmer in his book entitled "Tool Steel Simplified". Engineers and even metallurgists, however, unless they happen to have had first-hand experience (and far too few have had) do not seem to realize the dangers to most applications involved by using steels with coarse grain. Quality of toolsteels has been judged since time immemorial by observing the character of the fracture after quenching. About 20 years ago a set of fracture standards for grain size, ranging from No. 1 (coarse) to No. 10 (fine) was devised in Sweden by R. Arpi; B. F. Shepherd in America has done the same thing and stereoscopic photographs of his fracture standards have been published in *Metal Progress* for October 1943, page 594, and reproduced as Data Sheet No. 45 in the collected set. Austenitic grain size, determined under the microscope, has also been standardized by the American Society for Testing Materials.

Coming back again to the fracture grain size of toolsteel, many unnotched bar tests in a Charpy machine on carbon and carbon-vanadium toolsteels are summarized in Fig. 3. The results for high speed steel are practically identical. In fact, similar curves could be drawn for almost any kind of steel which is hardened. Note in particular how the steel really begins to "pay dividends" only above No. 9 fracture.

Yet how many of the present day A.I.S.I., NE, or S.A.E. steels meet this simple requirement without paying a sizable extra for specified grain size? If this situation were remedied, consumers would seldom be compelled to harden No. 5 or 6 fracture steels (as they are today) and take the risks and losses involved.

Fig. 3 — Effect of Fracture Grain Size on Transverse Strength in Bending and Charpy Test on Unnotched Bar, ¼x⅜x2 In., of 1% High Carbon Toolsteel, Hardened to Rockwell C-64

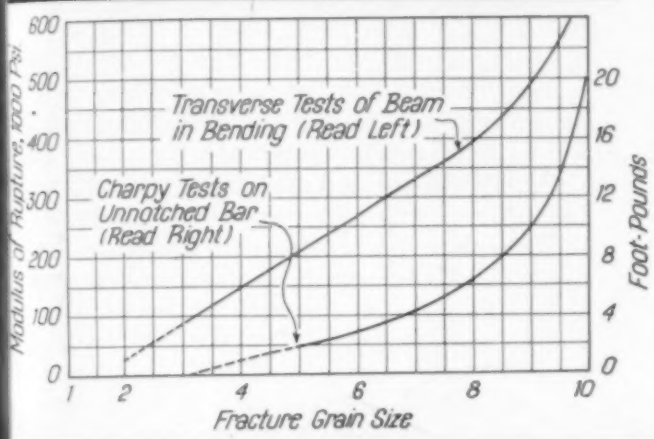


Figure 4 shows how coarse grain increases cracking of carbon toolsteel during quenching. The samples in this study must have been of an easy-hardening design, or the curve would have been displaced toward the right.

Coarse grain also increases the tendency to form grinding cracks. In fact, below No. 5 fracture the grains may spall out bodily, giving a pock-marked appearance to the surface. Fine grained chisel steels (No. 9 to 10) will develop a tensile strength of 350,000 psi. with 6% elongation and 16% reduction at Rockwell C-60 (700 Brinell), but when the fracture is only No. 7 to 8 the steel breaks "short", with less than 200,000 psi. tensile strength.

Having tested practically every type of toolsteel made here and in Europe, we are safe in saying that fine grain is more important than type of alloy combination, as far as strength and toughness are concerned.

Some years ago, the writer had occasion to investigate the failure of some ring gears made of S.A.E. 4140 and 4340 steel which had broken during assembly. Samples were cut from the pieces and the properties were as shown in Table I. Comparison values are shown for a similar composition, except that it contained vanadium and was fine grained.

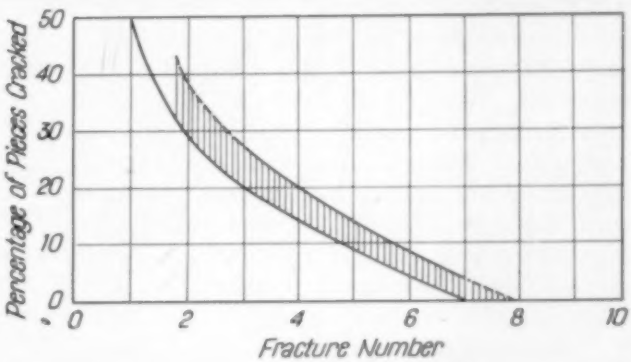


Fig. 4 — Grain Size of 1% Carbon Toolsteel Vs. Liability of Quenching Cracks. (R. Arpi; see Metal Progress, June 1931, p. 88)

The mere presence of vanadium will not guarantee fine fracture. Some of the worst steel we ever examined contained vanadium. On the other hand, the steels with the finest fracture (No. 9 or 10) and highest properties always contained vanadium; hence the conclusion is that vanadium makes good steel better.

General Vs. Special Purpose Steels — A general purpose steel is one which is suitable for a

Table I — Tests on Ring Gears of Three Steels

	S.A.E. 4140	S.A.E. 4340	Cr-Mo-Va
Fracture number	No. 4	No. 5	No. 9 to 10
Rockwell hardness	C-48 to 49	C-51	C-54
Tensile strength	226,000 psi.	244,000 psi.	288,000 psi.
Elastic limit	170,500 psi.	208,500 psi.	253,000 psi.
Reduction of area	36%	40%	63%
Charpy notched bar	2.5 ft-lb.	5 ft-lb.	16 ft-lb.

wide and diverse field of application. In lines of manufacture where mass production is not the rule (and there are many such) its availability would be more important than, say, a small differential in price.

Furthermore it seems conceivable that, at times, a special purpose steel may exceed a general purpose in tonnage demand. For example, say that there is a small part which is to be made in great quantities from steel of the class identified as Steel D in the matched set (page 726). This steel probably has more hardenability than the particular part would require, and therefore the user may find it economical to "shave" the alloy content, since he will take whole heats at one time. This, however, does not change the fundamental argument for simplification.

The trouble has been that too many steels of limited application have found their way into the S.A.E. specifications and other published lists. So they are given the stamp (unintentionally it may be) of official approval as general purpose materials. Such specialties should be confined to a private arrangement between user and seller. There is no objection to listing all sorts of steels in advertising literature, but it is senseless to clutter up the handbooks and official lists with every analysis that is made — at least, if we wish to achieve simplification.

Specification by Form — One of the most pernicious practices in the whole steel business has been that of issuing specifications according to some commercial form, condition, or finish, rather than by chemical analysis. After all, the chemical composition is the most basic and fundamental characteristic of any substance; it does not by itself determine all the properties, but it is the most important single thing that does.

Forging, welding, brazing, heat treatment, and other working procedures are pretty much the same for any given steel analysis, regardless of whether it is in the form of forgings, castings, bars, or tube. Why, then, should we write separate specifications for each? Truly this is confusing the form with the substance.

I know of one company which has as many as 18 separate specifications for what is essentially S.A.E. 1025, each with its slight variant of analysis. One was for castings, one for forging billets, another for forgings, one for bar and rod, two for wire, one each for structural shapes, wire rod, clamping rings, rivets, staybolts, and welding rods, and five for plate! This list does not include others which may have been issued by the scattered subsidiary works and plants; these may have numbered many more. Yet all these parts could have been made as well (and with improvement in most cases) with steel of the chemical composition used for castings.

Why should we have specifications for "spring" steel, when the same analysis which is good for springs (such as Steel B in the matched set) is good for many other uses?

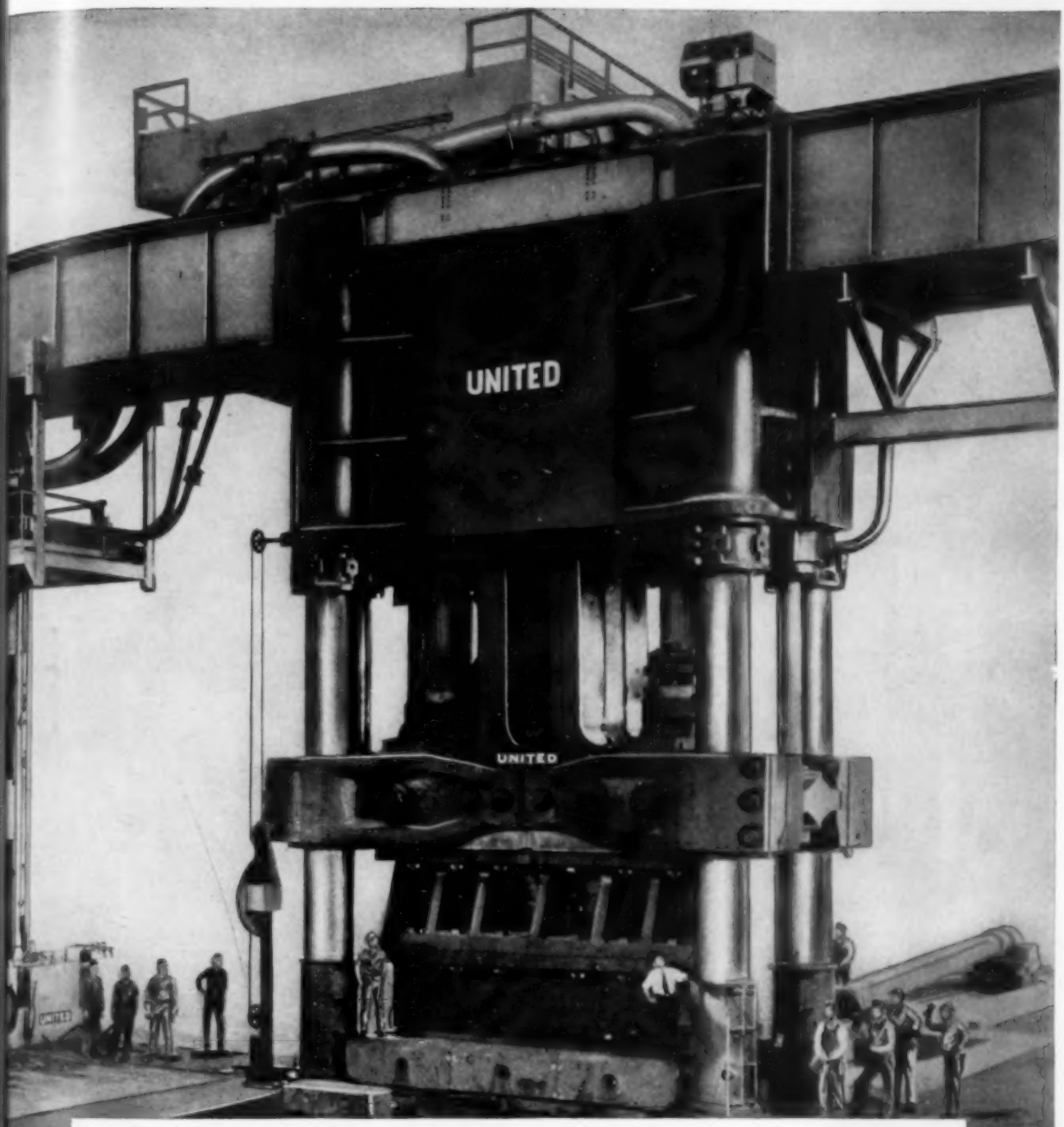
Even the plainest and mildest of the steels (or commercially pure iron, for that matter) can be strengthened enormously by cold plastic working, a fact made evident by the few simple calculations in Table II, made from tensile tests of annealed specimens in all cases. In this tabulation the "true" tensile strength is based upon the necked-down area at fracture. It is computed by dividing the "ultimate" tensile strength figured on the original area of the test specimen by the necked-down area. This figure may be taken as the theoretical maximum which may be approached, but not attained, by cold drawing.

Table II — True Tensile Strength of Plain Carbon Steels

	Ingot Iron	1020	1040	1095
"Ultimate" strength	40,000	64,000	87,000	105,000
Reduction of area	0.71	0.60	0.52	0.47
Relative area of reduced section at time of fracture	0.29	0.40	0.48	0.53
"True" tensile strength	138,000	160,000	181,000	198,000

Similar calculations for alloy steels show values as high as 300,000 psi. for the "true" tensile strength. Nickel and manganese seem particularly effective strengtheners of alloy steel after cold work.

Therefore, in conclusion, if we consider the overlapping properties obtainable by heat treatment or cold working, the steels listed ought to satisfy most of the requirements of any consumer, regardless of how large or diversified, and do it much better and more simply than it is being done today.



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With Notes on First Aid

Data Sheet by E. F. Houghton & Co.

MANY COMMERCIAL PREPARATIONS for use in the heat treatment of steel contain poisonous cyanide as an essential if not the principal ingredient. Their use is perfectly safe if simple precautions are taken, and has been so widespread that all heat treaters with any considerable background of experience are well acquainted with them, and recognize the hazards and how to avoid them. Newcomers in industry, however, must be properly and promptly instructed, in no uncertain terms; such instructions should be repeated until the supervisor is certain the workmen understand the dangers and how to avoid them.

Rules for Handling Salts

1. Use scoop and dry cotton gloves.
2. Do not handle cyanide if you have any broken skin or open wounds on hands or wrists.
3. Wash hands thoroughly after handling.
4. Keep the container covered when in storage.
5. Open the container only in the room or building in which the cyanide compound is used.
6. Keep cyanide salts away from acid. Cyanide in the presence of even the weakest acid will liberate the deadly gas known as hydrocyanic acid.
7. Food should not be stored, handled or eaten in the vicinity of cyanide mixtures.
8. Wear rubber gloves when operating the bath or handling articles which have passed through it.
9. In case any irritation of the skin develops as a result of handling cyanide, consult a physician immediately.
10. Burns from molten cyanide salt mixtures are treated as any burns caused by alkalies. Wash the area thoroughly with warm water to remove chemicals; then treat as any burn. If the burn is at all severe, see the doctor immediately.

Operating the Salt Bath

While not to be considered as a complete manual, observance of the seven suggestions below will avoid the common dangers to personnel operating salt baths.

1. Do not mix a compound containing cyanide with one containing nitrates or nitrites, or put one in a container which has been used for the other. Violent reaction and possible explosions result from such mixtures. Material treated in a bath of one sort should not be processed in a bath of the other until it has been thoroughly cleaned. Containers of the original salts are properly labeled so such mixing can be avoided. In case of doubt, don't mix.

2. Keep asbestos gloves always at hand.
3. Wear safety goggles or face mask when operating the bath.
4. Hoods and stacks are advisable for salt baths.
5. All work must be thoroughly dry before introduction, or splattering may result.
6. When bailing out a bath, molten salt should not be stored in a mold or container deeper than 6 in. The risk of serious eruption while solidifying increases with depth.
7. To avoid remelting hazards in all furnaces except those which heat from the top down (as the internal electrode type) follow this procedure: Before the bath freezes, insert a cast iron wedge with tip touching the bottom while the thick end projects at least 4 in. above the surface. The vent remaining when the wedge is removed from the solidified salt will permit the escape of gases on remelting. Remove the wedge by tapping with a hammer, but not until the bath is completely solidified — if removed earlier, molten salt below the surface may erupt.

First Aid for Cyanide Poisoning

First aid is the emergency treatment to prevent further harm or possible death. It is applied immediately, prior to the arrival of physician.

The first requirement is to obtain from a druggist and always have on hand for instant use in the first aid cabinet *in the heat treat:*

Amyl nitrite pearls (heart stimulant).

Sodium thiosulphate, 1% solution (an emetic, to induce vomiting and empty the stomach).

Call physician immediately; meanwhile apply first aid treatment promptly as follows:

If patient is conscious and breathing

1. Break an amyl nitrite pearl in a piece of cotton gauze and hold lightly over his nose while the patient inhales.
2. If cyanide has been swallowed, induce vomiting by giving a pint of sodium thiosulphate, 1% solution. If this is not ready at hand (which is inexcusable) give copious drinks of soapy water, mustard water, or bicarbonate of soda (2 teaspoons to pint of water). Continue the use of any of these emetics until the patient throws up a clear solution, which proves his stomach is empty.

If patient is unconscious but breathing

1. Break an amyl nitrite pearl in a piece of cotton gauze and hold lightly over his nose while the patient inhales.
2. Follow with oxygen from an inhalator, if it is available.
3. Give nothing by mouth to an *unconscious* man.

If patient is not breathing

1. Give artificial respiration at once, continuing until physician arrives.
2. While artificial respiration proceeds, an assistant breaks an amyl nitrite pearl in a piece of cotton gauze and holds it to the patient's nose.
3. Give nothing by mouth to an *unconscious* patient.

ELECTROLYTIC POLISHING OF STAINLESS STEEL AND OTHER METALS

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ANODIC METHODS of electrolytically cleaning metals have been known for a long time. It was early recognized that when the electrolyte's temperature and concentration were properly regulated bright cleaned surfaces were obtained. However, it was not until 1935, when the original paper of Pierre Jacquet was published (Reference 8 in the Bibliography, page 735), that the subject of electrolytic polishing received the attention of the metallurgical world. It has since been the object of much interest.

Electrolytic polishing of many metals and alloys has been developed by metallographers for preparing specimens for microscopic examination. Its application to some metals, such as aluminum and copper, has been concerned with the obtaining of surfaces with higher reflecting power than those obtained by mechanical polishing. It is in the field of stainless steel, however, that electrolytic polishing has attained industrial importance.

A lustrous finish on stainless steel requires difficult and costly grinding and buffing operations. Electrolytic polishing obtains a mirrorlike finish without the use of any such mechanical methods and at a much lower cost. Mechanical polishing of stainless steel excessively cold works the surface into a highly stressed, partly ferritic condition, in which the metal is flowed and piled into the scratch troughs. Furthermore, particles of abrasive are frequently imbedded — a condition which tends to promote pit corrosion. Because of the poor thermal conductivity and hardness of stainless steel, mechanical polishing heats the surface in a very non-uniform manner.

Electrolytically polished surface layers, on the other hand, are free of stress (or have the same stress as existed in the metal before polishing), are free of foreign particles, contain no ferrite, and have no heat-affected regions. Moreover, the surface is passivated. Such a surface has greater corrosion resistance than has a mechanically polished surface.

Formed articles which are particularly difficult to polish, mechanically, because of inaccessible or depressed areas are easily polished by electrolysis. For example, a complex welded assembly can be readily polished electrolytically at a fraction of the cost of doing it mechanically.

All types of stainless steels and irons can be polished by this chemical means.

This paper primarily presents the various solutions reported to be satisfactory for the polishing of stainless steel, and discusses some of the variables involved in obtaining a bright surface from anodic action. The literature contains information on useful electrolytes for other metals, and this information is also collected in a supplementary table (page 734).

Essentially, the process consists in making the article to be polished the anode in an acid electrolytic bath capable of forming a soluble salt with the constituent metal. The cathode can be

stainless steel, lead, graphite, duriron, or other conductive, inactive material, about 10 to 20 times the size of the anode (the work being polished). Incidentally, where many objects or parts of the same intricate shape are to be polished, conforming cathodes that have the same general shape as the parts should be used. This promotes uniform smoothness down into the recesses.

The current density should be sufficient to form and to maintain a solid oxidation product on the anode (the work), and permit it to have a viscous flow from the surface of the anode. This film is thicker in the troughs of the scratches than it is on the peaks. Consequently, the resistance to dissolution is less at a high point of a scratch than it is at a lower point; the peak dissolves faster and eventually a plane surface is reached.

Since the mechanism removes the high spots there will be necessarily a size loss. Brightening can be obtained with a loss of 0.0005 in. from the surface, but a satisfactory polish will generally remove more metal than this — say, from 0.001 to 0.002 in. Another use of electrolytic polishing technique is suggested, and that is the removal of excess surface. When a rod or bar is definitely oversize by a small amount, electrolytic polishing can be satisfactorily used to correct the condition. Furthermore, since the metal dissolves most rapidly at the edges, burrs can be readily removed; this is of especial use, of course, when the burrs are inaccessible to a wheel.

Two Classes of Solutions

The solutions for electrolytic polishing may be placed in either of two categories. The first is composed of those solutions of low electrical resistance, requiring low voltage of from 4 to 25 volts, wherein control of polishing comes from voltage control. The second includes solutions of high electrical resistance, requiring high voltage of from 50 to 220 volts, wherein control of polishing comes from current control.

In the first category are the solutions based upon phosphoric acid and acids such as sulphuric, arsenic, chromic, boric, tartaric, citric, acetic, malic, succinic, and lactic, with or without certain organic addition reagents as glycerol, alcohol, ether, carbitol, methyl cellosolve, ethylene glycol, benzoic acid, picric acid, commercial inhibitors, benzene and toluene sulphonates. These organic addition agents affect the conductivity of the anode film and retard localized pitting.

In the second category are those solutions based upon perchloric acid and organic reagents like acetic acid, acetic anhydride, alcohol, and ether. Solutions in both categories contain small proportions of water to improve the conductivity.

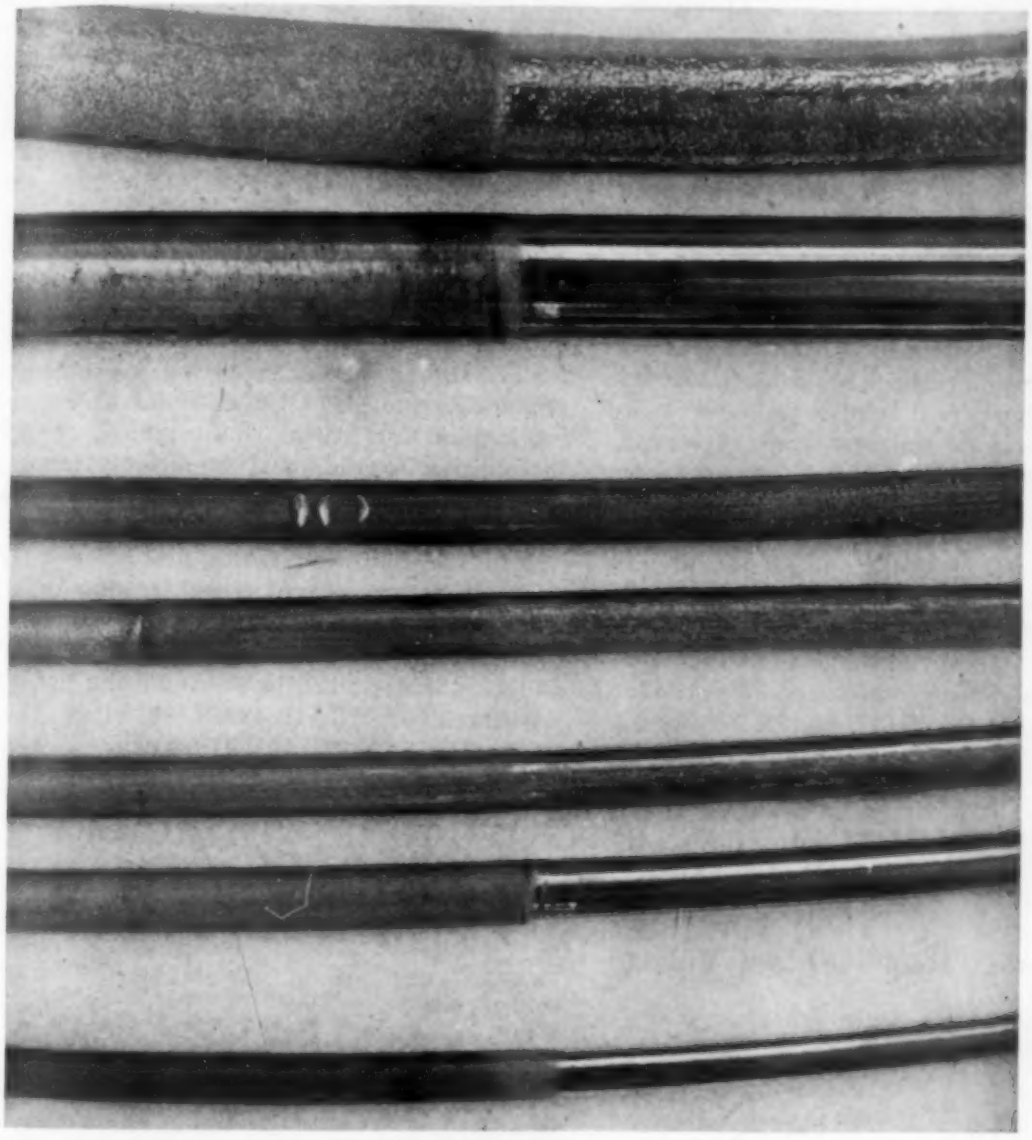
The current density required for polishing varies according to the solution used, in either category, and ranges from a few tenths of an ampere per sq.in. to 30 or even 50 amperes per sq.in. A "critical" value exists below which polishing cannot occur regardless of the time allowed, because the film does not form and the specimen is etched. Increasing the current density above the critical value for polishing shortens the time required. Because of greater metal loss and lessened chances of pitting, the higher current densities yield more lustrous surfaces. The anode current efficiencies of the solutions range from 25 to 100%. Good polishing solutions have good throwing power.

Those solutions of the first category should be operated hot for best performance, at about 100 to 300° F., depending on the solution. The higher the temperature the higher the current density required. Second category solutions should be operated cold (temperature not exceeding 85° F.). Since the current input will heat the solution, external cooling is necessary. Tanks can be lead lined, or made of stainless steel.

The article to be polished should be totally immersed, as the foaming in some solutions results in a poor polish on that portion within the foam; furthermore, attack on the metal at the solution line is severe. The distance between the anode and the cathode may be from 0.5 to 5.0 in. When the work is coiled (wire or strip) polishing is best done continuously, with the strand being drawn through the polishing solution, wash, and dryer.

Proper surface preparation is a pre-requisite for a satisfactory polish. Rough surfaces, however, can be brightened. For metallographic specimens the surface should first be taken down mechanically to the finish produced by No. 0 paper. The smoother the surface is to begin with, the less the polishing time. The use of very high current densities shortens the polishing time markedly and also reduces the amount of preparatory surface treatment required.

If wire is to be formed into articles to be subsequently electrolytically polished, skilful draw bench practice must give the wire a smooth and uniform surface. Minor defects that are difficult to detect in the drawn wire and stress irregularities that are impossible to see become quite apparent in the surface after the wire has been electrolytically polished. Any forming operation must be carried out so that no highly localized, highly stressed areas are present. If they are unavoidable it is necessary to remove


Brightened Hot-Rolled Surface
Brightened Ground Surface
Pitted From HCl Pickle
Effect of Strained Surface on Polishing
Polished After Strained Surface Removed
Polished Stainless Iron

Samples of Stainless Steel Wires; One End of Each Has Been Polished or Brightened by an Electrolytic Process

the stressed surfaces or the stresses before polishing. As a corollary of this, electrolytic polishing affords an excellent inspection method for seams, heavy carbide precipitation, highly stressed areas, and heterogeneity in wire and strip.

In addition to stress effects on the surface, the internal condition of the steel — that is, the size and distribution of the inclusions, and the homogeneity of the structure — affects the final polish. Inclusions are always dissolved out; consequently a specimen electrolytically polished for microscopic examination will appear dirtier than when mechanically polished. Agitation of the solution is not necessary for large scale polishing but it does reduce pitting around the inclusions, and aids in preparing microscope specimens.

Polished surfaces may be streaked by gas bubbles streaming upward. High temperatures, addition agents and agitation will help to avoid this trouble. Agitation increases the anode efficiency and thus the dissolution of the specimen is more rapid in those areas near the greatest turbulence. Consequently, agitation is undesirable when polishing flat surfaces.

Distorted metal at the surface of mechanically polished stainless steel is completely absent when the steel is electrolytically polished. Microscopic examination of an electrolytically polished

surface shows no scratch marks, stress bands, or other evidences of a distorted structure. An electrodeposited metal film on an electrolytically polished and etched specimen of stainless steel has a grain structure that is a continuance of the grain structure of the base metal, while a similar plate on a mechanically polished and etched specimen shows no such structure because it is deposited on the distorted surface layer.

Perchloric acid solutions entail a certain hazard. This hazard, the noxious fuming, and the fact that perchloric acid is a more expensive reagent than phosphoric acid, has caused industry to adopt phosphoric acid solutions almost exclusively for polishing the stainless steels. (There is, however, at least one large installation based on perchloric acid.) Metallographic samples to be polished in perchloric acid should be removed from their plastic, or Wood's metal mounts, as these materials form explosive compounds with the reagents. Apparatus for laboratory polishing is described in some detail in each of the following literature references to be found on page 735: 22, 23, 28, 40, 44, 45, 65, 67, 80, 84, 85, 88, 89, 93, 107, 110.

Phosphoric acid solutions have a long life (several years), and can be operated over a wide range of compositions. Any one of the solutions listed will give good results. Patented modifications will brighten an article in shorter times, and in some instances permit brighter surfaces to be obtained than from the simple phosphoric acid solutions in water.

Post-war applications of electrolytic polishing for improving the appearance of stainless steel are many, but the process merits more application now than it is receiving. Correction for oversize, deburring, removing amorphous layers, scale removal, polishing of intricate shapes, preparation for plating, improvement of finish, in addition to the most important feature of increasing the corrosion resistance, are good reasons for this statement.

In the following tables are presented the various solutions recommended for stainless steel and for other metals, along with the sources of the recommendations. Apparatus for commercial polishing is described in the following literature references (page 735): 12, 24, 29, 54, 56, 57, 58, 60, 62, 71, 76, 26, 99, 101, 113, 114.

A SURVEY OF SOLUTIONS REPORTED TO BE SATISFACTORY FOR POLISHING STAINLESS STEEL

Part A: Solutions Requiring Low Voltage

(All solutions are given in *weight* percentages unless otherwise indicated; current in amperes per sq.in.; temperatures in °F.)

Solution		Conditions	Reference
Phosphoric acid	100	1 amp. 100-220° 5 min.	4, 76, 78
Phosphoric acid Water	80 20	1-12 amp. 100-220° 5 min.	4, 29
Phosphoric acid Water	75 25	3 amp. 100-220° 5 min.	4, 25
Pyrophosphoric acid Alcohol to make 1 liter	400 g.	2 amp. 100-220° 10 min.	42, 47, 85
Sulphuric acid Aqueous solution	75–100	20 amp. 150° 5 min.	1, 2, 38, 76, 78, 112
Chromic acid Water	43 57	0.4 amp. 100° 1 hr.	6
Phosphoric acid Sulphuric acid Water	60 20 20	5 amp. 175° 3 min.	91
Phosphoric acid Sulphuric acid Water	65 15 20	3.5 amp. 200° 5 min.	76, 113
Phosphoric acid Sulphuric acid Water	63 15 22	0.35 amp. 120° 1 hr.	97, 98
Phosphoric acid Sulphuric acid Water	30 60 10	0.2 amp. 120° 5 min.	76, 97
Phosphoric acid Sulphuric acid Water	30 60 10	1.8 amp. 120° 2 min.	98
Phosphoric acid Sulphuric acid Water	45 40 15	0.5-3.5 amp. 85-300° 5 min.	76
Phosphoric acid Sulphuric acid Nitric acid	14 50 36	No current 120° 5 min.	7
Phosphoric acid Sulphuric acid Chromic acid Water	63 15 10 12	3 amp. 120° 1 hr.	48, 106
Phosphoric acid Sulphuric acid Chromic acid Water	67 20 4 9	3 amp. 120° 1 hr.	106
Phosphoric acid Sulphuric acid Chromic acid Water	15 60 10 15	4 amp. 120° 30 min.	106
Phosphoric acid Sulphuric acid Chromic acid Water	67 20 2 11	1.7 amp. 120° 1 hr.	98, 106

Solution		Conditions	Reference
Phosphoric acid	44	3 amp.	98, 106
Sulphuric acid	40	120°	
Chromic acid	6	1 hr.	
Water	10		
Phosphoric acid	13	7 amp.	97
Sulphuric acid	16	120°	
Glycerol	56	5 min.	
Water	15		
Phosphoric acid	42	0.1-0.6 amp.	51, 101,
Glycerol	47	200-300°	104, 75
Water	11	8-15 min.	
(Can also make other organic additions, which are high boiling, soluble in phosphoric acid, and contain one or more hydroxyl groups)			
Phosphoric acid	40	0.5 amp.	35
Glycerol, tallow, benzoic acid, picric acid, or inhibitors	50	212° 10 min.	
Water	10		
Phosphoric acid	50	3-4 amp.	109
Sulphuric acid	40	160°	
Solution A, containing 7 lb. glycine dissolved in 1 gallon hot water	10	2-3 min.	
Phosphoric acid	70	0.7-3.5 amp.	108
Chromic acid	10	100°	
Water	20	5 min.	
Phosphoric acid	60	1.8 amp.	83
Arsenic acid	15	120-160°	
Water	20	40 min.	
Sulphuric acid	48	3.5 amp.	35
Glycerol	32	130°	
Water	20	5 min.	
Sulphuric acid	58	1-2 amp.	52
Glycerol	20	70-100°	
Water	22	5 min.	
Sulphuric acid	73	1.5 amp.	74
Glycerol or Toluene sulphonates	20	85° 0.5-5 min.	
Water	7		
Sulphuric acid	50	7 amp.	97
Glycerol	40	180°	
Water	10	8 min.	
Sulphuric acid	60	14 amp.	97
Ammoniated-glycyrrhizin	0.01	165° 2 min.	
Methanol	0.55		
Water	40		
Sulphuric acid	15	0.5-40 amp.	96
Citric acid	60	125-250°	
Water	25	0.5-3 min.	
Add 4% of total volume of methyl alcohol, or other alcohols as ethyl, propyl, butyl, glycerol, glycol			
Sulphuric acid	20	0.5-1.5 amp.	56, 62
Citric acid	55	180-190°	
Water	25	5 min.	
Sulphuric acid	15	0.5-40 amp.	49, 99
Citric acid	55	120-250°	
Water	30	0.5-3 min.	
Sulphuric acid	10	0.3-0.7 amp.	83
Arsenic acid	77	140°	
Water	13	20-90 min.	
Sulphuric acid	25	0.3-0.7 amp.	82, 83
Arsenic acid	69	140°	
Water	6	20-90 min.	
Sulphuric acid	10	0.7 amp.	82
Arsenic acid	77	140°	
Water	13	60 min.	
Sulphuric acid	36	0.3-0.7amp.	82
Arsenic acid	47	140°	
Water	17	60 min.	
Sulphuric acid	55	0.5-2 amp.	24
Hydrofluoric acid	7	70°	
Water	38	0.5-4 min.	
Sulphuric acid	69	2 amp.	24
Hydrofluoric acid	10	70°	
Hydrogen peroxide	21	5 min.	
Sulphuric acid	73	3.5 amp.	24
Hydrofluoric acid	13	70°	
Water	14	5 min.	
Sulphuric acid	73	1 amp.	13
Hydrofluoric acid	7	140°	
Water	20	5 min.	
Sulphuric acid	40	Specimen as cathode	39
Hydrochloric acid	29.5		
Titanium tetrachloride	5.5	170°	
Nitric acid	0.5	2-5 min.	
Water	24.5		
Sulphuric acid	17	1 amp.	3
Calcium phosphate, phosphoric acid added		75°	
Sodium sulphate added to saturation		1.5 min. as anode 1.5 min. as cathode	
Arsenic acid	84	2.5 amp.	83
Chromic acid	10	140°	
Water	6	10 min.	
Aqueous acid electrolytic bath containing any of the following ions that form easily soluble salts with the steel: Oxalate, bisulphite, chloride, nitrate, bromide, cyanate, sulphite, acetate, fluoride, iodate, iodide, perchlorate, nitrate, chlorate, thiocyanate, bromate, hydrosulphide, and cyanide.		Current density sufficient to remove solid anodic oxidation products from the surface. 50-100°	50

Part B: Solutions Requiring High Voltage

(All solutions are given in *volume* percentages. Current in amperes per sq.in.; temperatures in °F.)

Solution		Conditions	Reference
Perchloric acid	75	2-4 amp.	81
Water	25	50-85° 5 min.	
Perchloric acid	32	0.4 amp.	100, 34
Acetic anhydride	64	85° max.	
Water	4	5 min.	
Perchloric acid	19	0.6-4 amp.	91, 32,
Acetic anhydride	76	85° max.	30, 34,
Water	5	4-10 min.	66
Perchloric acid	34	0.5 amp.	54, 61,
Acetic anhydride	66	85° max. 5 min.	45
Perchloric acid	20	13-40 amp.	77, 72,
Ethyl alcohol	80	95° max. 10-15 sec.	73, 79, 93
Perchloric acid	66	30 amp.	92
Ethyl alcohol	80	85° max.	
Water	14	30 sec.	
Nitric acid	34	10 amp.	45
Methyl alcohol	66	90-100° 1 min.	

A SURVEY OF SOLUTIONS REPORTED TO BE SATISFACTORY FOR POLISHING SEVERAL METALS AND ALLOYS

Aluminum

Solution		Conditions	Reference
Perchloric acid	20 (vol.)	0.2-0.4 amp.	20, 61
Acetic anhydride	80	100° max.	
		15 min.	
Perchloric acid	20 (vol.)	13-26 amp.	72
Ethyl alcohol with 3 per cent ether	80	95° max.	
		8-12 sec.	
Dilute aqueous solution of fluoboric acid			27, 63
Sodium carbonate or phosphate aqueous solution			16
Chromate or phosphate solution			70

Copper

Phosphoric acid	63 (wt.)	0.02-0.05 amp.	8, 10, 84, 61
Water	37	68°	
		5 min.	
Phosphoric acid	70 (wt.)	0.4-0.5	
Ether	30	or 3-4.5 amp.	5, 10
		80°	
		5-10 min.	
Pyrophosphoric acid	58 (wt.)	0.5-0.7 amp.	61, 68
Water	42	68°	
		10-15 min.	

Brass

Chromic acid	16 (wt.)	60-80 amp.	95
Water	84		
Phosphoric acid	70 (wt.)	0.5 amp.	61
Water	30	80°	
		5 min.	

Lead

Perchloric acid	30 (vol.)	1.3-1.6 amp.	61
Glacial acetic acid	70	68°	
		5 min.	
Perchloric acid	24 (vol.)	1-2.5 amp.	61
Acetic anhydride	67	68°	
Water	9	5 min.	
Perchloric acid	35 (vol.)	0.6-0.7 amp.	61
Acetic anhydride	63	68°	
Water	2	5 min.	

Nickel

Sulphuric acid	73 (wt.)	1-3 amp.	37, 52
Water	27	80°	
		2 min.	
Sulphuric acid	70 (wt.)	2-4 amp.	18
Water	30	100°; 5 min.	
Perchloric acid	75 (vol.)	2-4 amp.	5
Glacial acetic acid	25	85°	
		5 min.	

Steel; Carbon and Alloy

Annealed			
Perchloric acid	18.5 (vol.)	0.3-0.8 amp.	34, 61,
Acetic anhydride	76.5	68°	80, 107
Water	5.0	4-5 min.	
Quenched			
Perchloric acid	20 (vol.)	13-32 amp.	72
Ethyl alcohol with 3 per cent ether	80	68°	
		10-15 sec.	
Tempered Steel			
Phosphoric acid	50 (vol.)	0.5-1 amp.	51
Diethylene glycol monoethyl ether	50	120-175°	
		15 min.	
Electrical Sheet with 3 per cent Silicon			
Perchloric acid	33.5 (vol.)	0.4 amp.	34
Glacial acetic acid	66.5	85° max.	
		5 min.	
Hadfield's Manganese			
Perchloric acid	18 (vol.)	2 amp.	66
Acetic anhydride	76	85° max.	
Water	6	30 sec.	
General Application on Steels			
Perchloric acid	10 (vol.)	1.5-2.5 amp.	107
Glacial acetic acid	90	80°	
		0.5-2 min.	
Phosphoric acid	48 (wt.)	5-8 amp.	80
Sulphuric acid	40	95-120°	
Water	12	10 min.	
Phosphoric acid	45 (wt.)	3-7 amp.	80
Sulphuric acid	39	100°	
Glycerol	7	5-10 min.	
Water	9		
Phosphoric acid	46 (wt.)	1.5-4.5 amp.	80
Sulphuric acid	40	80-100°	
Dextrose	4	5-10 min.	
Water	10		
Phosphoric acid	65 (wt.)	3-7 amp.	80
Sulphuric acid	15	100-130°	
Chromic acid	5	3-5 min.	
Water	15		
Phosphoric acid	60 (wt.)	5 amp.	91
Sulphuric acid	20	175°	
Water	20	3 min.	

Tin

Perchloric acid	20 (vol.)	0.6-1 amp.	31
Acetic anhydride	80	85°	
		8-10 min.	

Tungsten

Caustic soda, aqueous solution, 10%		0.2-0.4 amp.	33, 61
		68°	
		20-30 min.	
Tri-sodium phosphate	160 g.	0.6 amp.	
Water	1 l.	100-120°	
		10 min.	

Zinc

Caustic potash aqueous 25% solution		1 amp.	23, 61
		68°	
		15 min.	
Chromic acid	17 (wt.)	16 amp.	95
Water	83		
Chromic acid	7 (wt.)	2 amp.	111
Sodium dichromate	22		
Sulphuric acid	6		
Acetic acid	7		
Water	58		

Miscellaneous Metals

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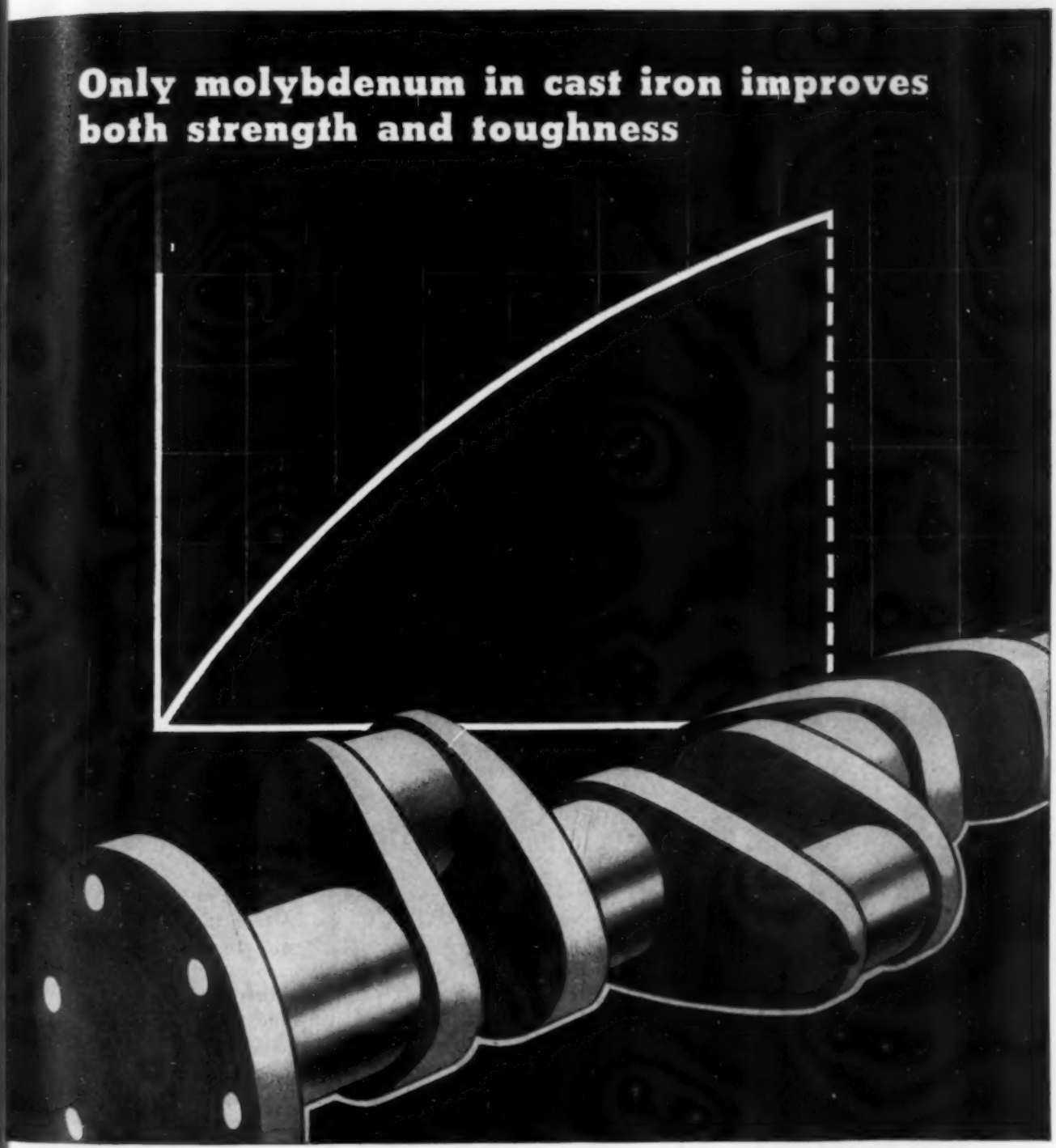

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PERSONALS

ROBERT S. MULLER has joined the staff of the American Society for Metals, *Metal Progress*, and *The Metals Review* as eastern representative with offices in New York City. CHESTER L. WELLS, formerly eastern representative, has taken up duties on the business staff in Cleveland.

PHILIP A. KOERNER is now employed as metallurgical engineer by Beech Aircraft Corp., Wichita, Kansas.

J. BJORKSTEN has left his position as chemical director of the Quaker Chemical Products Corp. to establish an independent industrial research organization in Chicago.

ENSIGN J. TUCKER MACKENZIE U.S.N. has received his wings and reported to Daytona Beach, Fla., U. S. Naval Air Station, for further training.

ARTHUR L. LAMASTERS, formerly general manager and chief engineer of the Claud S. Gordon Co., Chicago, is now vice-president in charge of operations of the Alloy Casting Co., Champaign, Ill.

R. E. FRITSCH, formerly vice-president, has been elected president of Tube Turns, Louisville, Ky.

HENRY T. CHANDLER, formerly assistant to president, has been elected a vice-president of the Vanadium Corp. of America. BENTON H. GRANT, formerly assistant to president, has been appointed an assistant vice-president.

R. E. ZIMMERMAN, vice-president, U. S. Steel Corp., has been appointed to a committee of eight industrial executives to advise the Department of Commerce and the American Standards Association on future plans for standards work.

JAMES H. BALDREY has been appointed superintendent of the melting department of Allegheny Ludlum Steel Corp., Watervliet, N. Y., succeeding T. F. MCCLESTER, who is retiring.

J. RAYMOND SMITH, formerly manager of stainless steel sheet sales for the Eastern Stainless Steel Corp., has been appointed to the newly created position of assistant to general sales manager of Rustless Iron and Steel Corp., Baltimore.

IVER G. FREEMAN, formerly superintendent of the Grinding Machine Division of Norton Co., Worcester, Mass., has been appointed factory manager.

JOE HOEFER has returned to the Udylite Corp., Detroit, to become sales and service engineer for the Michigan territory, succeeding BERT LUPIEN, now manager of the Cleveland territory.

WARREN J. MEAD, formerly head of the geology department of Massachusetts Institute of Technology, has been appointed director of all research activities for Reynolds Metals Co. REID B. GRAY, formerly director of the Glenn L. Martin Co.'s testing laboratories, has been named director of development research, and RICHARD J. LUND, until recently head of the Miscellaneous Minerals Division of the W.P.B., is in charge of basic research for Reynolds.

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PERSONALS

FRED E. STANLEY, formerly with the Eastern Aircraft Corp., Linden Division of General Motors Corp., Linden, N. J., is now metallurgist in the Research Laboratory, Engineering Department, Consolidated Vultee Aircraft Corp., Fort Worth, Texas.

Promoted by W. M. Chace Co., Detroit: CLARENCE F. ALBAN from chief metallurgist to chief engineer.

STANLEY D. WHITESIDE has been appointed district engineer for the Michigan area for W. S. Rockwell Co., New York.

W. E. RENWICK is now associated with the Mansfield Brass Foundry, Inc., Mansfield, Ohio, following dissolution of the Graphite Section of the W.P.B., of which he had been chief.

HAROLD R. KING has been appointed metallurgist of McCallum Hatch Bronze Co., Buffalo, N. Y.

HENRY M. PFAHL, formerly with Carnegie-Illinois Steel Corp. as contact metallurgist, is now with Tate-Jones & Co., Inc., Leetsdale, Pa., as metallurgical engineer and assistant manager of sales.

H. P. ETTER, formerly manager of the Los Angeles district for Air Reduction Co., has been appointed sales manager of the Pacific Coast Division with headquarters in San Francisco.

ARTHUR H. HJORTSBERG, formerly division superintendent of central rolling mills for Carnegie-Illinois Steel Corp., Gary Steel Works, has been made assistant operating head of the plant, succeeding ERLE G. HILL, now superintendent of Wheeling Steel Corp., Wheeling, West Va. JOHN J. GOLDEN has been promoted from superintendent of the steel production division of the Gary Works to division superintendent of the combined openhearths and central mills. EDWIN H. GOTT, former assistant division superintendent of maintenance, is promoted to assistant to general superintendent.

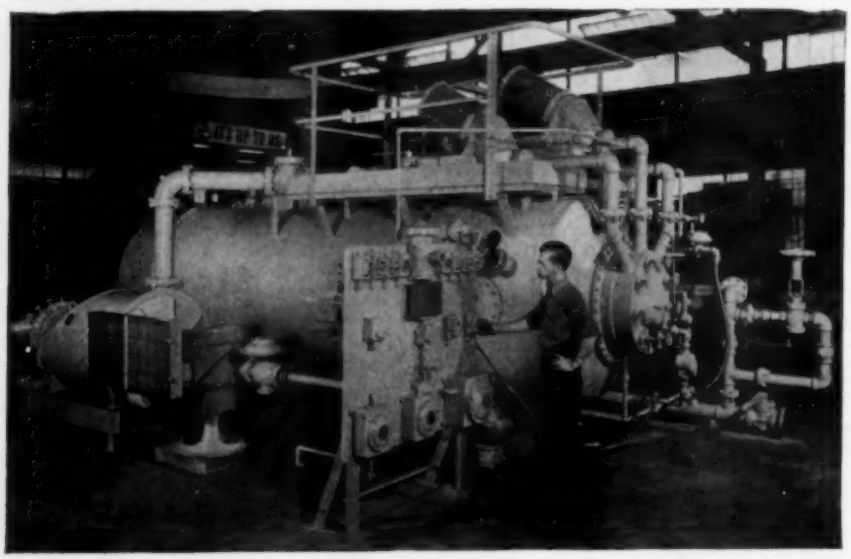
WM. H. COLVIN, JR., formerly president of Rotary Electric Steel Co., is now president of Crucible Steel Co. of America.

ROBERT E. BOCKRATH has been made manager of magnesium sales for the Houston, Texas office of the Dow Chemical Co.

WILLIAM H. SAFRANEK, JR., formerly chemist at the Apollo Metal Works, Clearing, Ill., has been appointed to the Staff of Battelle Memorial Institute, Columbus, Ohio, where he will engage in electrochemical research.

N. R. ARANT, formerly helper at Republic Steel Corp., South Chicago, is now junior melter in the electrical furnace shop at Universal-Cyclops Steel Corp., Bridgeville, Pa.

JOHN L. GIOVE is now employed at the Homestead Steel Works of Carnegie-Illinois Steel Corp. as junior metallurgist in research and development of armor plate.



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